EXHIBIT 1

US005782707A

United States Patent [19]

Yamagishi et al.

[11] Patent Number: 5,782,707

[45] Date of Patent: Jul. 21, 1998

[54] **THREE-PIECE SOLID GOLF BALL**

[75] Inventors: **Hisashi Yamagishi; Hiroshi Higuchi**, both of Chichibu, Japan

[73] Assignee: **Bridgestone Sports Co., Ltd.**, Tokyo, Japan

[21] Appl. No.: **812,925**

[22] Filed: **Mar. 10, 1997**

[30] **Foreign Application Priority Data**

Mar. 11, 1996 [JP] Japan 8-082121

[51] **Int. Cl.**^{6} **A63B 37/06**; A63B 37/12; A63B 37/14

[52] **U.S. Cl.** **473/374**; 473/373

[58] **Field of Search** 473/373, 374, 473/378, 384

[56] **References Cited**

U.S. PATENT DOCUMENTS

4,431,193	2/1984	Nesbitt	473/374
5,048,838	9/1991	Chikaraishi	473/373 X
5,273,286	12/1993	Sun	473/376 X
5,439,227	8/1995	Egashira et al.	473/377 X
5,601,503	2/1997	Yamagishi et al.	473/351 X
5,688,191	11/1997	Cavallaro et al.	473/373

Primary Examiner—George J. Marlo
Attorney, Agent, or Firm—Sughrue, Mion, Zinn, Macpeak & Seas, PLLC

[57] **ABSTRACT**

The invention provides a three-piece solid golf ball featuring an increased flight distance on driver shots and improved control on approach shots. In a three-piece solid golf ball consisting of a solid core, an intermediate layer, and a cover, provided that hardness is measured by a JIS-C scale hardness meter, the core center hardness is up to 75 degrees, the core surface hardness is up to 85 degrees, the core surface hardness is higher than the core center hardness by 8 to 20 degrees, the intermediate layer hardness is higher than the core surface hardness by at least 5 degrees, and the cover hardness is lower than the intermediate layer hardness by at least 5 degrees.

6 Claims, 2 Drawing Sheets

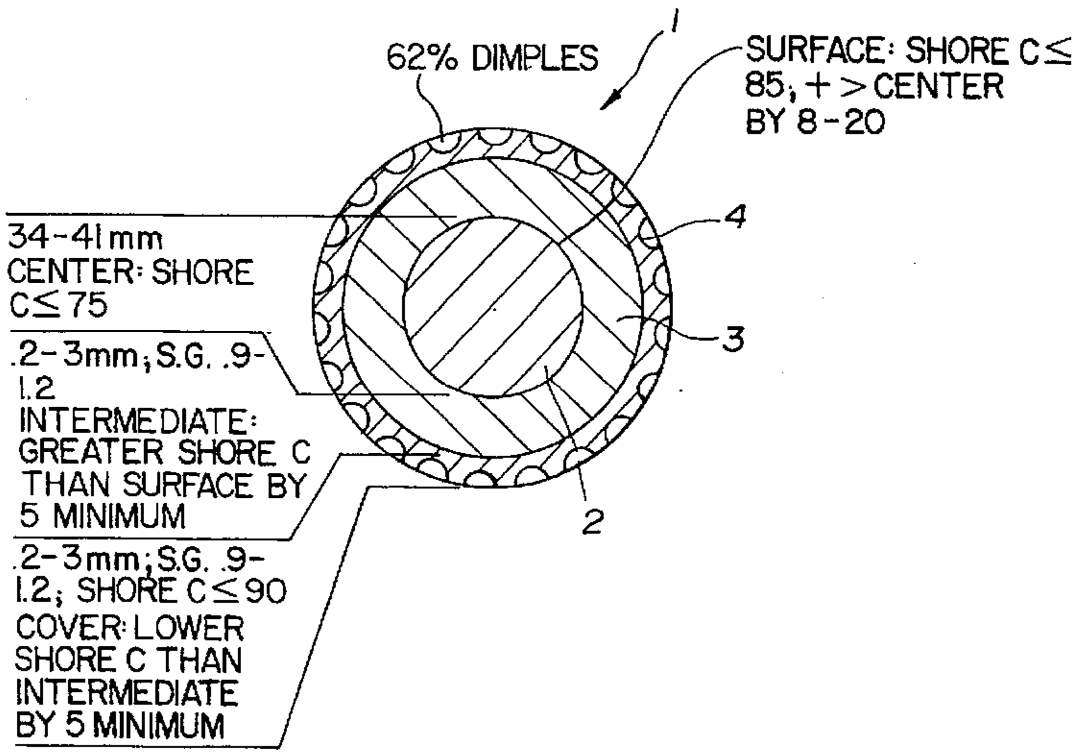


FIG.1

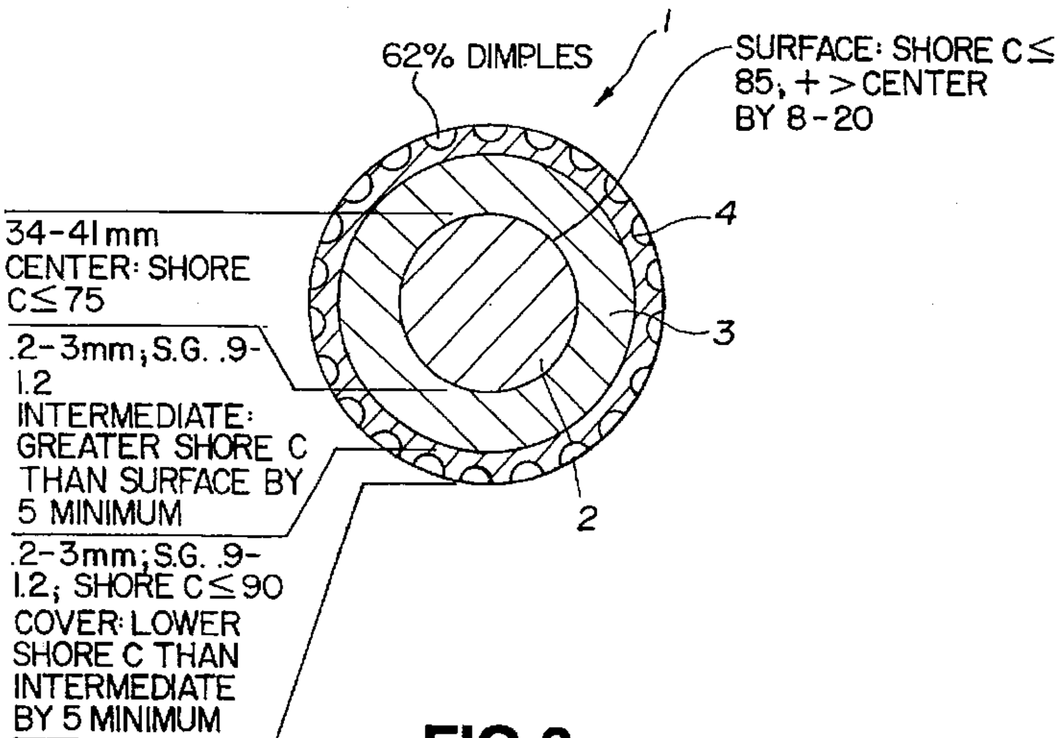

62% DIMPLES
1
SURFACE: SHORE C≤ 85; + > CENTER BY 8-20
4
3
2
34-41mm CENTER: SHORE C≤75
.2-3mm; S.G. .9-1.2 INTERMEDIATE: GREATER SHORE C THAN SURFACE BY 5 MINIMUM
.2-3mm; S.G. .9-1.2; SHORE C≤90 COVER: LOWER SHORE C THAN INTERMEDIATE BY 5 MINIMUM

FIG.2

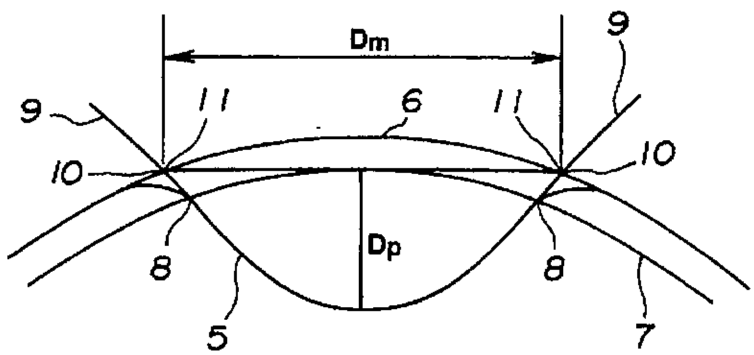

Dm
9
11
6
11
9
10
10
Dp
8
8
5
7

FIG.3

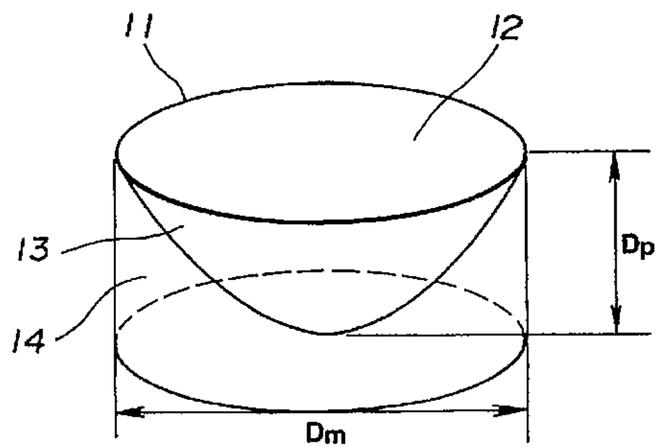
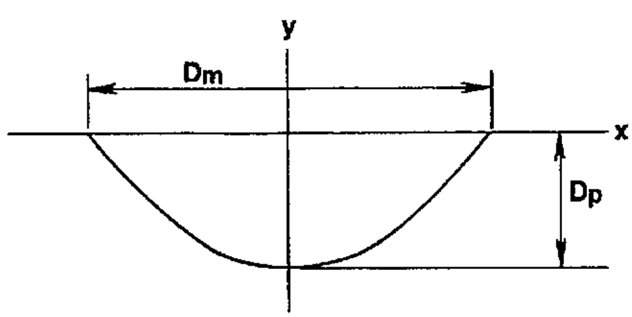


FIG.4



THREE-PIECE SOLID GOLF BALL

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to a three-piece solid golf ball of the three-layer structure comprising a solid core, an intermediate layer, and a cover and more particularly, to such a three-piece solid golf ball which features an increased flight distance on full shots with a driver and improved control on approach shots with No. 5 iron or sand wedge.

2. Prior Art

From the past, two-piece solid golf balls consisting of a solid core and a cover are used by many golfers because of their flight distance and durability features. In general, two-piece solid golf balls give hard hitting feel as compared with wound golf balls, and are inferior in feel and control due to quick separation from the club head. For this reason, many professional golfers and skilled amateur golfers who prefer feel and control use wound golf balls rather than two-piece solid golf balls. The wound golf balls are, however, inferior in carry and durability to the solid golf balls.

More particularly, when two-piece solid golf balls are subject to full shots with a club having a relatively large loft angle, the ball flight is mainly governed by the club loft rather than the ball itself so that spin acts on most balls to prevent the balls from too much rolling. However, on approach shots over a short distance of 30 to 50 yards, rolling or control substantially differs among balls. The major cause of this difference is not related to the basic structure of the ball, but to the cover material. Then some two-piece solid golf balls use a cover of a relatively soft material in order to improve control on approach shots, but at the sacrifice of flight distance.

Controllability is also needed on full shots with a driver. If a soft cover is used as a result of considering too much the purpose of improving spin properties upon control shots such as approach shots with No. 5 iron and sand wedge, hitting the ball with a driver, which falls within an increased deformation region, will impart too much spin so that the ball may fly too high, resulting in a rather reduced flight distance. On the other hand, if the spin rate is too low, there arises a problem that the ball on the descending course will prematurely drop, adversely affecting the ultimate flight distance too. As a consequence, an appropriate spin rate is still necessary upon driver shots.

Anyway, the prior art two-piece solid golf balls fail to fully meet the contradictory demands of players, the satisfactory flight performance that the ball acquires an adequate spin rate upon full shots with a driver and the ease of control that the ball acquires a high spin rate upon approach shots with No. 5 iron and sand wedge.

SUMMARY OF THE INVENTION

An object of the present invention is to provide a three-piece solid golf ball which features an increased flight distance on full shots with a driver and improved control on approach shots with No. 5 iron or sand wedge.

Making extensive investigations on a three-piece solid golf ball of the three-layer structure comprising a solid core, an intermediate layer, and a cover, we have found that the above object is attained by optimizing the hardness distribution of the core, forming a hard intermediate layer between the core and the soft cover, and adjusting a percent dimple surface occupation. By virtue of the synergistic effect of these factors, the resulting golf ball travels an increased flight distance on full shots with a driver and is well controllable on approach shots with No. 5 iron or sand wedge.

More specifically, we have found that the following advantages are obtained in a three-piece solid golf ball of the three-layer structure comprising a solid core, an intermediate layer, and a cover, when the solid core, intermediate layer, and cover each have a hardness as measured by a JIS-C scale hardness meter, the core center hardness is up to 75 degrees, the core surface hardness is up to 85 degrees, the core surface hardness is higher than the core center hardness by 8 to 20 degrees, the intermediate layer hardness is higher than the core surface hardness by at least 5 degrees, and the cover hardness is lower than the intermediate layer hardness by at least 5 degrees. Upon deformation in an increased deformation region (associated with full shots with a driver), the presence of a hard intermediate layer between a soft deformable cover and a soft core ensuring soft feel is effective for reducing the energy loss by excessive deformation of the core and thereby enabling to form a structure of efficient restitution while maintaining the softness of the ball as a whole. Then the ball will travel an increased flight distance upon full shots with a driver. Although a soft cover is used, the ball gains an appropriate spin rate and is free of shortage of flight distance. At the same time, in a reduced deformation region (associated with approach shots), the ball gains an increased spin rate and is well controllable. Additionally, by adjusting dimples such that the percent surface occupation of dimples in the cover surface is at least 62% and an index (Dst) of overall dimple surface area is at least 4, and optimizing the dimple pattern, the flight properties (flight distance and flight-in-wind) of the golf ball are further enhanced. By virtue of the synergistic effect of these factors, the resulting golf ball covers an increased flight distance on full shots with a driver and is well controllable on approach shots with No. 5 iron or sand wedge, that is, satisfies the contradictory demands of players.

Therefore, according to the present invention, there is provided a three-piece solid golf ball of the three-layer structure comprising a solid core, an intermediate layer, and a cover, having a plurality of dimples in the ball surface. Provided that the solid core at its surface and center, the intermediate layer, and the cover each have a hardness as measured by a JIS-C scale hardness meter, the core center hardness is up to 75 degrees, the core surface hardness is up to 85 degrees, the core surface hardness is higher than the core center hardness by 8 to 20 degrees, the intermediate layer hardness is higher than the core surface hardness by at least 5 degrees, and the cover hardness is lower than the intermediate layer hardness by at least 5 degrees. The dimples occupy at least 62% of the ball surface.

In one preferred embodiment, the dimples in the ball surface total in number to 360 to 450 and include at least two types of dimples having different diameters. An index (Dst) of overall dimple surface area given by the following expression (1) is at least 4.

$$Dst = \frac{\pi \sum_{k=1}^{n} [(Dmk^2 + Dpk^2) \times V_0 k \times Nk]}{4R^2} \qquad (1)$$

wherein R is a ball radius, n is the number of dimple types, Dmk is a diameter of dimples k, Dpk is a depth of dimples k, Nk is the number of dimples k wherein k=1, 2, 3, . . . n, and V_0 is the volume of the dimple space below a plane circumscribed by the dimple edge divided by the volume of a cylinder whose bottom is the plane and whose height is the maximum depth of the dimple from the bottom.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic cross-sectional view of a three-piece solid golf ball according to one embodiment of the invention.

FIG. 2 is a schematic cross-sectional view of a dimple illustrating how to calculate V_0.

FIG. 3 is a perspective view of the same dimple.

FIG. 4 is a cross-sectional view of the same dimple.

DETAILED DESCRIPTION OF THE INVENTION

Referring to FIG. 1, a three-piece solid golf ball 1 according to the invention is illustrated as comprising a solid core 2 having an optimized hardness distribution, a hard intermediate layer 3, and a soft cover 4.

In the golf ball 1 of the invention, the hardness distribution of the solid core 2 is optimized. More particularly, the core 2 is formed to have a center hardness of up to 75 degrees, preferably 60 to 73 degrees, more preferably 63 to 69 degrees as measured by a JIS-C scale hardness meter. The core 2 is also formed to have a surface hardness of up to 85 degrees, preferably 70 to 83 degrees, more preferably 73 to 80 degrees. If the core center hardness exceeds 75 degrees and the surface hardness exceeds 85 degrees, the hitting feel becomes hard, contradicting the object of the invention. It is noted that the hardness referred to herein is JIS-C scale hardness unless otherwise stated.

The core is formed herein such that the surface hardness is higher than the center hardness by 8 to 20 degrees, preferably 10 to 18 degrees. A hardness difference of less than 8 degrees would result in a hard hitting feel provided that the ball hardness and the core surface hardness are fixed. A hardness difference of more than 20 degrees would fail to provide sufficient restitution provided that the ball hardness and the core surface hardness are fixed. The hardness distribution establishing such a hardness difference between the surface and the center of the core ensures that the core surface formed harder than the core center is effective for preventing excessive deformation of the core and efficiently converting distortion energy into reaction energy when the ball is deformed upon impact. Additionally, a pleasant feeling is obtainable from the core center softer than the core surface.

The hardness distribution of the solid core is not limited insofar as the core is formed such that the core surface is harder than the core center by 8 to 20 degrees. It is preferable from the standpoint of efficient energy transfer that the core is formed such that the core becomes gradually softer from its surface toward its center.

The solid core preferably has a diameter of 34 to 41 mm, especially 34.5 to 40 mm. No particular limit is imposed on the overall hardness, weight and specific gravity of the core and they are suitably adjusted insofar as the objects of the invention are attainable. Usually, the core has an overall hardness corresponding to a distortion of 2.5 to 4.5 mm, especially 2.8 to 4 mm under a load of 100 kg applied, and a weight of 20 to 40 grams, especially 23 to 37 grams.

In the practice of the invention, no particular limit is imposed on the core-forming composition from which the solid core is formed. The solid core may be formed using a base rubber, a crosslinking agent, a co-crosslinking agent, and an inert filler as used in the formation of conventional solid cores. The base rubber used herein may be natural rubber and/or synthetic rubber conventionally used in solid golf balls although 1,4-cis-polybutadiene having at least 40% of cis-structure is especially preferred in the invention. The polybutadiene may be blended with a suitable amount of natural rubber, polyisoprene rubber, styrenebutadiene rubber or the like if desired. The crosslinking agent includes organic peroxides such as dicumyl peroxide, di-t-butyl peroxide, and 1,1-bis(t-butylperoxy)-3,3,5-trimethylcyclohexane, with a blend of dicumyl peroxide and 1,1-bis(t-butylperoxy)-3,3,5-trimethylcyclohexane being preferred. In order to form a solid core so as to have the above-defined hardness distribution, it is preferable to use a blend of dicumyl peroxide and 1,1-bis(t-butylperoxy)-3,3,5-trimethylcyclohexane as the crosslinking agent and the step of vulcanizing at 160° C. for 20 minutes. It is noted that the amount of the crosslinking agent blended is suitably determined although it is usually about 0.5 to 3 parts by weight per 100 parts by weight of the base rubber. The co-crosslinking agent used herein is not critical. Examples include metal salts of unsaturated fatty acids, inter alia, zinc and magnesium salts of unsaturated fatty acids having 3 to 8 carbon atoms (e.g., acrylic acid and methacrylic acid), with zinc acrylate being especially preferred. Examples of the inert filler include zinc oxide, barium sulfate, silica, calcium carbonate, and zinc carbonate, with zinc oxide and barium sulfate being often used. The amount of the filler blended is usually up to 40 parts by weight per 100 parts by weight of the base rubber although the amount largely varies with the specific gravity of the core and cover, the standard weight of the ball, and other factors and is not critical. In the practice of the invention, the overall hardness and weight of the core can be adjusted to optimum values by properly adjusting the amounts of the crosslinking agent and filler (typically zinc oxide and barium sulfate) blended.

The core-forming composition obtained by blending the above-mentioned components is generally milled in a conventional mixer such as a Banbury mixer and roll mill, compression or injection molded in a core mold, and then heat cured under the above-mentioned temperature condition, whereby a solid core having an optimum hardness distribution is obtainable.

The intermediate layer 3 enclosing the core 2 is preferably formed to a JIS-C hardness of 75 to 100 degrees, more preferably 80 to 98 degrees. The intermediate layer is formed to a hardness higher than the core surface hardness by at least 5 degrees, preferably 5 to 20 degrees, more preferably by 7 to 18 degrees. A hardness difference of less than 5 degrees would fail to provide sufficient restitution whereas a hardness difference of more than 20 degrees would result in a dull and rather hard hitting feel. The restitution of the core can be maintained by forming the intermediate layer to a higher hardness than the core surface hardness.

The gage, specific gravity and other parameters of the intermediate layer may be properly adjusted insofar as the objects of the invention are attainable. Preferably the gage is 0.2 to 3 mm, especially 0.7 to 2.3 mm and the specific gravity is 0.9 to less than 1.2, especially 0.94 to 1.15.

Since the intermediate layer 3 serves to compensate for a loss of restitution of the solid core which is formed soft, it is formed of a material having improved restitution insofar as a hardness within the above-defined range is achievable. Use is preferably made of a blend of ionomer resins such as Himilan (manufactured by Mitsui-duPont Polychemical K.K.) and Surlyn (E.I. duPont) as will be described later in Table 2. An intermediate layer-forming composition may be obtained by adding to the ionomer resin, additives, for example, an inorganic filler such as zinc oxide and barium sulfate as a weight adjuster and a coloring agent such as titanium dioxide.

The cover 4 enclosing the intermediate layer 3 must be formed to a lower hardness than the intermediate layer. That is, the cover has a hardness lower than the intermediate layer hardness by at least 5 degrees. Additionally, the cover is preferably formed to a JIS-C hardness of up to 90 degrees, more preferably 70 to 90 degrees, most preferably 75 to 87 degrees when spin properties in an approach range are of much account. A cover hardness in excess of 90 degrees on JIS-C scale would adversely affect the spin properties in an approach range so that professional and skilled amateur players who prefer accurate control reject use in the game. A cover hardness of less than 70 degrees would result in a ball losing restitution.

The gage, specific gravity and other parameters of the cover may be properly adjusted insofar as the objects of the invention are attainable. Preferably the gage is 0.2 to 3 mm, especially 0.7 to 2.3 mm and the specific gravity is 0.9 to less than 1.2, especially 0.93 to 1.15. The gage of the intermediate layer and cover combined is preferably 2 to 4.5 mm, especially 2.2 to 4.2 mm.

The cover composition is not critical and the cover may be formed of any of well-known stock materials having appropriate properties as golf ball cover stocks. For example, ionomer resins, polyester elastomers, and polyamide elastomers may be used alone or in admixture with urethane resins and ethylene-vinyl acetate copolymers. Thermoplastic resin base compositions are especially preferred. UV absorbers, antioxidants and dispersing aids such as metal soaps may be added to the cover composition if necessary. The method of applying the cover is not critical. The cover is generally formed over the core by surrounding the core by a pair of preformed hemispherical cups followed by heat compression molding or by injection molding the cover composition over the core.

Like conventional golf balls, the three-piece solid golf ball of the invention is formed with a multiplicity of dimples in the cover surface. The golf ball of the invention is formed with dimples such that, provided that the golf ball is a sphere defining a phantom spherical surface, the proportion of the surface area of the phantom spherical surface delimited by the edge of respective dimples relative to the overall surface area of the phantom spherical surface, that is the percent occupation of the ball surface by the dimples is at least 62%, preferably 63 to 85%. With a dimple occupation of less than 62%, the above-mentioned flight performance, especially an increased flight distance is not expectable. The total number of dimples is preferably 360 to 450, more preferably 370 to 440. There may be two or more types of dimples which are different in diameter and/or depth. It is preferred that the dimples have a diameter of 2.2 to 4.5 mm and a depth of 0.12 to 0.23 mm. The arrangement of dimples may be selected from regular octahedral, dodecahedral, and icosahedral arrangements as in conventional golf balls while the pattern formed by thus arranged dimples may be any of square, hexagon, pentagon, and triangle patterns.

Moreover, the dimples are preferably formed such that V_0 is 0.39 to 0.6, especially 0.41 to 0.58 wherein V_0 is the volume of the dimple space below a plane circumscribed by the dimple edge divided by the volume of a cylinder whose bottom is the plane and whose height is the maximum depth of the dimple from the bottom.

Now the shape of dimples is described in further detail. In the event that the planar shape of a dimple is circular, as shown in FIG. 2, a phantom sphere 6 having the ball diameter and another phantom sphere 7 having a diameter smaller by 0.16 mm than the ball diameter are drawn in conjunction with a dimple 5. The circumference of the other sphere 7 intersects with the dimple 5 at a point 8. A tangent 9 at intersection 8 intersects with the phantom sphere 6 at a point 10 while a series of intersections 6 define a dimple edge 11. The dimple edge 11 is so defined for the reason that otherwise, the exact position of the dimple edge cannot be determined because the actual edge of the dimple 5 is rounded. The dimple edge 11 circumscribes a plane 12 (having a diameter Dm). Then as shown in FIGS. 3 and 4, the dimple space 13 located below the plane 12 has a volume Vp. A cylinder 14 whose bottom is the plane 12 and whose height is the maximum depth Dp of the dimple from the bottom or circular plane 12 has a volume Vq. The ratio V_0 of the dimple space volume Vp to the cylinder volume Vq is calculated.

$$Vp = \int_0^{\frac{Dm}{2}} 2\pi xy dx$$

$$Vq = \frac{\pi Dm^2 Dp}{4}$$

$$V_0 = \frac{Vp}{Vq}$$

In the event that the planar shape of a dimple is not circular, the maximum diameter or length of a dimple is determined, the plane projected shape of the dimple is assumed to be a circle having a diameter equal to this maximum diameter or length, and V_0 is calculated as above based on this assumption.

Furthermore, provided that the number of types of dimples formed in the ball surface is $\underline{n}$ wherein $n \geqq 2$, preferably n=2 to 6, more preferably n=3 to 5, and the respective types of dimples have a diameter Dmk, a maximum depth Dpk, and a number Nk wherein k=1, 2, 3, . . . , n, the golf ball of the invention prefers that an index Dst of overall dimple surface area given by the following equation (1) is at least 4, more preferably 4 to 8.

$$Dst = \frac{n \sum_{k-1}^{n} [(Dmk^2 + Dpk^2) x V_0 k x Nk]}{4R^2} \qquad (1)$$

Note that R is a ball radius, V_0 is as defined above, and Nk is the number of dimples k. The index Dst of overall dimple surface area is useful in optimizing various dimple parameters so as to allow the golf ball of the invention having the above-mentioned solid core and cover to travel a further distance. When the index Dst of overall dimple surface area is equal to or greater than 4, the aerodynamics (flying distance and flight-in-wind) of the golf ball are further enhanced.

While the three-piece solid golf ball of the invention is constructed as mentioned above, other ball parameters including weight and diameter are properly determined in accordance with the Rules of Golf.

The three-piece solid golf ball of the invention will travel an increased flight distance on full shots with a driver and be easy to control on approach shots with No. 5 iron or sand wedge.

EXAMPLE

Examples of the present invention are given below together with Comparative Examples by way of illustration and not by way of limitation. The amounts of components in the core, intermediate layer, and cover as reported in Tables 1 and 2 are all parts by weight.

Examples 1–5 and Comparative Examples 1–4

Solid cores, Nos. 1 to 6, were prepared by kneading components in the formulation shown in Table 1 to form a rubber composition and molding and vulcanizing it in a mold under conditions as shown in Table 1. The cores were measured for JIS-C hardness and diameter, with the results shown in Tables 3 and 4. The JIS-C hardness of the core was measured by cutting the core into halves, and measuring the hardness at the center (center hardness) and the hardness at core surface or spherical surface (surface hardness). The result is an average of five measurements.

TABLE 1

Core No.	1	2	3	4	5	6
Formulation						
Cis-1,4-poly-butadiene rubber	100	100	100	100	100	100
Zinc acrylate	24	24	25	29	15	34
Zinc oxide	29	26	34	27	33	25
Dicumyl peroxide	1	1	1	1	1	0
*1	0.3	0.3	0.3	0.3	0.3	1
Vulcanizing conditions						
Temperature, °C.	160	160	160	160	160	155
Time, min.	20	20	20	20	20	15
Core hardness*2, mm	3.7	3.7	3.5	3	5.7	2.2

*1 1,1-bis(t-butylperoxy)-3,3,5-trimethylcyclohexane (trade name Perhexa 3M-40 manufactured by Nippon Oil and Fats K.K.)
*2 distortion under a load of 100 kg

Next, compositions for the intermediate layer and cover were milled as shown in Table 2 and injection molded over the solid core and the intermediate layer, respectively, obtaining three-piece solid golf balls as shown in Table 4. At the same time as injection molding, two or three types of dimples were indented in the cover surface as shown in Table 3. Whenever the intermediate layer and cover were molded, the intermediate layer and cover were measured for JIS-C hardness, specific gravity and gage. The results are also shown in Table 4.

TABLE 2

Intermediate layer and cover formulations (pbw)					
	A	B	C	D	E
Himilan 1557*3	50	—	50	—	—
Himilan 1601*3	—	—	50	—	—
Himilan 1605*3	50	50	—	—	—
Himilan 1855*3	—	—	—	50	50
Himilan 1856*3	—	—	—	—	50
Himilan 1706*3	—	50	—	—	—
Surlyn 8120*4	—	—	—	50	—

*3 ionomer resin manufactured by Mitsui-duPont Polychemical K.K.
*4 ionomer resin manufactured by E.I. duPont of USA

TABLE 3

Dimple set	Diameter (mm)	Depth (mm)	V_0		Number	Dst	Surface occupation (%)
I	4.000	0.200	0.50		72	4.539	75
	3.850	0.193	0.50		200		
	3.400	0.170	0.50		120		
				total	392		
II	3.800	0.205	0.48		162	4.263	74
	3.600	0.194	0.48		86		
	3.450	0.186	0.48		162		
				total	410		
III	3.400	0.195	0.39		360	2.148	61
	2.450	0.195	0.39		140		
				total	500		

The thus obtained golf balls were evaluated for flight performance, spin, feel, spin control, and durability by the following tests.

Flight performance

Using a hitting machine manufactured by True Temper Co., the ball was actually hit with a driver (#W1) at a head speed of 45 m/s (HS45) and 35 m/sec. (HS35) to measure a spin, carry, and total distance.

Feel

Five golfers with a head speed of 45 m/sec. (HS45) and five golfers with a head speed of 35 m/sec. (HS35) actually hit the balls. The ball was rated according to the following criterion.

O:soft

Δ:ordinary

X:hard

Spin control

Three professional golfers actually hit the ball with No. 5 iron (#I5) to examine intentional hook and slice and stoppage on the green and also with a sand wedge (#SW) to examine spin on 30 and 80 yard shots (that is, stoppage on the green and ease of capture of the ball upon impact). An overall rating of the ball was derived from these spin control factors. The ball was rated "O" for easy control, "Δ" for ordinary, and "X" for difficult control.

Durability

Durability against continuous strikes and durability against cutting were evaluated in combination. The ball was rated according to the following criterion.

O:excellent

Δ:ordinary

X:inferior

TABLE 4

	Examples					Comparative Examples			
	1	2	3	4	5	1	2	3	4
Core									
Type	1	2	3	4	1	1	5	6	4
Center hardness A (JIS-C)	64	64	65	68	64	64	52	80	68
Surface hardness B (JIS-C)	75	75	77	82	75	75	62	90	82
B − A	11	11	12	14	11	11	10	10	14
Diameter (mm)	36.5	37.9	35.1	37.9	36.5	36.5	36.5	36.5	37.9
Intermediate layer									
Type	A	A	B	B	C	A	D	B	A
Hardness C (JIS-C)	86	86	93	93	83	86	75	93	86
C − B	11	11	16	11	8	11	13	3	4
Specific gravity	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97
Gage (mm)	1.6	1.2	1.8	1.2	1.6	1.6	1.6	1.6	1.8
Cover									
Type	E	E	C	F	D	E	B	A	B
Hardness D (JIS-C)	80	80	83	80	75	81	93	86	93
D − C	−6	−6	−10	−13	−8	−5	18	−7	7
Specific gravity	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97
Gage (mm)	1.5	1.5	2.0	1.5	1.5	1.5	1.5	3.5	2.0
Intermediate layer/cover combined gage (mm)	3.1	2.7	3.8	2.7	3.1	3.1	3.1	5.1	3.8
Dimple set	I	I	II	II	II	III	I	I	I
Ball outer diameter (mm)	42.7	42.7	42.7	42.7	42.7	42.7	42.7	42.7	42.7
#W1/HS45									
Spin (rpm)	2800	2750	2900	2700	2950	2800	2650	2700	2680
Carry (m)	209.0	210.0	210.0	209.5	210.5	207.0	209.0	207.5	208.5
Total (m)	223.0	224.5	223.5	222.0	224.0	218.0	221.0	217.0	218.0
Feel	O	O	O	O	O	O	Δ	X	X
#W1/HS35									
Spin (rpm)	4600	4400	4650	4700	4750	4600	4600	4680	4630
Carry (m)	142.0	144.0	142.5	144.0	143.0	138.0	142.5	139.0	140.0
Total (m)	150.0	153.0	150.0	152.5	152.0	145.0	149.5	145.5	148.0
Feel	O	O	O	O	Δ	O	Δ	X	X
Spin control	O	O	O	O	O	O	X	Δ	X
Durability	O	O	O	O	O	O	X	Δ	Δ

Note:
A hardness difference is represented by (B − A), (C − B), and (D − C). (B − A) is equal to the core surface hardness minus the core center hardness; (C − B) is equal to the intermediate layer hardness minus the core surface hardness; and (D − C) is equal to the cover hardness minus the intermediate layer hardness.

As is evident from Table 4, the ball of Comparative Example 1 which is identical with the ball of Example 1 except for the dimple set is unsatisfactory in flight distance because the dimple surface occupation is as low as 61%. The ball of Comparative Example 2 is inferior in hitting feel, spin control, and durability since the cover is harder than the intermediate layer. The ball of Comparative Example 3 is unsatisfactory in flight distance and hitting feel because the core surface hardness and core center hardness are too high and the hardness difference between the intermediate layer and the core surface is too small. The ball of Comparative Example 4 is inferior in flight distance, hitting feel, and spin control since the cover is harder than the intermediate layer and the intermediate layer is insufficiently harder than the core.

In contrast, the golf balls of Examples 1 to 5 within the scope of the invention receive an appropriate spin rate upon full shots with a driver to travel a longer flight distance, are easy to spin control upon approach shots, and are excellent in both hitting feel and durability.

Japanese Patent Application No. 82121/1996 is incorporated herein by reference.

Although some preferred embodiments have been described, many modifications and variations may be made thereto in the light of the above teachings. It is therefore to be understood that within the scope of the appended claims, the invention may be practiced otherwise than as specifically described.

We claim:

1. A three-piece solid golf ball of the three-layer structure comprising a solid core, an intermediate layer, and a cover, having a plurality of dimples in the ball surface wherein

the solid core, intermediate layer, and cover each have a hardness as measured by a JIS-C scale hardness meter wherein the core center hardness is up to 75 degrees, the core surface hardness is up to 85 degrees, the core surface hardness is higher than the core center hardness by 8 to 20 degrees, the intermediate layer hardness is higher than the core surface hardness by at least 5 degrees, and the cover hardness is lower than the intermediate layer hardness by at least 5 degrees, and

the dimples occupy at least 62% of the ball surface.

2. The three-piece solid golf ball of claim 1 wherein said intermediate layer has a gage of 0.2 to 3 mm and a specific gravity of 0.9 to less than 1.2.

3. The three-piece solid golf ball of claim 1 wherein said cover is based on a thermoplastic resin and has a hardness of up to 90 degrees as measured by the JIS-C scale hardness meter.

4. The three-piece solid golf ball of claim 1 wherein said cover has a gage of 0.2 to 3 mm and a specific gravity of 0.9 to less than 1.2.

5. The three-piece solid golf ball of claim 1 wherein said solid core is formed of a cis-1,4-polybutadiene base elastomer and has a diameter of 34 to 41 mm.

6. The three-piece solid golf ball of claim 1 wherein the dimples in the ball surface total in number to 360 to 450 and include at least two types of dimples having different

diameters, and an index (Dst) of overall dimple surface area given by the following expression is at least 4,

$$Dst = \frac{n \sum_{k-1}^{n} [(Dmk^2 + Dpk^2) x V_0 k x Nk]}{4R^2}$$

wherein R is a ball radius, n is the number of dimple types ($n \geqq 2$), Dmk is a diameter of dimples k, Dpk is a depth of dimples k, Nk is the number of dimples k wherein k=1, 2, 3, . . . n, and V_0, is the volume of the dimple space below a plane circumscribed by the dimple edge divided by the volume of a cylinder whose bottom is the plane and whose height is the maximum depth of the dimple from the bottom.

* * * * *

UNITED STATES PATENT AND TRADEMARK OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 5,782,707 Page 1 of 2
DATED : July 21, 1998
INVENTOR(S) : Hisashi Yamagishi et al.

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

Please add claims 7-17 as follows:

7. The three-piece solid golf ball of claim 6 wherein Dmk is in the range of 2.2 to 4.5 and Dpk is in the range of 0.12 to 0.23 mm.

8. The three-piece solid golf ball of claim 6 wherein V_0 is in the range of 0.39 to 0.6.

9. The three-piece solid golf ball of claim 1 wherein said core center hardness is in the range of 60 to 73 as measured on JIS-C.

10. The three-piece solid golf ball of claim 1 wherein said core has a surface hardness in the range of 70 to 83 degrees on JIS-C.

11. The three-piece solid golf ball of claim 1 wherein said core surface hardness is higher than the center hardness by 10 to 18 degrees.

12. The three-piece solid golf ball of claim 1 wherein said solid core has a distortion in the range of 2.5 to 4.5 mm under an applied load of 100 kg.

13. The three-piece solid golf ball of claim 1 wherein said intermediate layer has a hardness in the range of 75 to 100 degrees measured on JIS-C.

14 The three-piece solid golf ball of claim 1 wherein said intermediate layer has a hardness higher than the core surface hardness by t to 20 degrees.

15. The three-piece solid golf ball of claim 1 wherein said cover has a hardness in the range of 70 to 90 degrees measured on JIS-C.

UNITED STATES PATENT AND TRADEMARK OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 5,782,707
DATED : July 21, 1998
INVENTOR(S) : Hisashi Yamagishi et al.

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

16. The three-piece solid golf ball of claim 1 wherein the gage of the intermediate layer and the cover combined is in the range of 2 to 4.5 mm.

17. The three-piece solid golf ball of claim 1 wherein said dimples occupy 63 to 85% of the ball surface

Signed and Sealed this

Sixth Day of November, 2001

Attest:

Nicholas P. Godici

NICHOLAS P. GODICI

Attesting Officer

Acting Director of the United States Patent and Trademark Office

EXHIBIT 2

REDACTED

EXHIBIT 3

UNITED STATES DISTRICT COURT
DISTRICT OF DELAWARE

BRIDGESTONE SPORTS CO., LTD., and BRIDGESTONE GOLF, INC., Plaintiffs, v. ACUSHNET COMPANY, Defendant.	Case No. 05-CA-132 (JJF) **INVALIDITY EXPERT REPORT OF DR. DAVID FELKER**
ACUSHNET COMPANY, Counterclaimant, v. BRIDGESTONE SPORTS CO., LTD., and BRIDGESTONE GOLF, INC., Counterdefendant.	

(where the ball distortion is slightly less than the core distortion). Through an understanding of the basic physics of ball construction and/or knowledge of the Precept EV Extra Spin golf ball, one of ordinary skill in the art of golf ball manufacturing would appreciate that the ratio of core distortion divided by ball distortion should be between 1.0 and 1.3, as they did for the similar-constructions used by the Precept EV Extra Spin golf ball and in the GB '628 publication.

2. The Claimed Range of Shore D Hardness Would Have Been Obvious To One Of Ordinary Skill In The Art

As further shown by Bridgestone's own Precept EV Extra Spin golf ball, it would have been obvious to use a cover with a Shore D hardness of up to 60 with a soft core construction. The soft core is designed to improve feel; a cover with a Shore D hardness of up to 60 contributes to the feel, whereas a harder cover would detract from it. This fact was recognized in the Precept EV Extra Spin, which used a cover with a Shore D hardness of 52.

VII. THE '707, '834, AND '791 PATENTS

A. Overview of the Patents

I will now address three related Bridgestone patents: U.S. Patent No. 5,782,707 (the '707 Patent) (Ex. 25), U.S. Patent No. 5,803,834 (the '834 Patent) (Ex. 26) and U.S. Patent No. 6,6791,791 (the '791 Patent) (Ex. 27). These three patents claim golf balls having a core with a surface harder than the center, where the hardness of the core increases radially outward from the center, sometimes increasing by a specific amount. This feature is referred to in the art as a hardness gradient in the core. In particular, a core's "hardness gradient" is a measurement of how the hardness of the core's rubber changes from the center of the core to its surface.

1. The '707 Patent

The '707 patent, entitled a "Three-Piece Solid Golf Ball," was applied for at the PTO on March 10, 1997, claiming priority to Japanese patent application no. 8-082121, filed March 11, 1996. The PTO issued the '707 Patent to Bridgestone on July 21, 1998, naming as the inventors Hisashi Yamagishi and Hiroshi Higuchi. The claims of the '707 are directed to the following

combination of three elements: (1) a core with a particular hardness distribution, whose key feature is a hardness gradient of 8-20 degrees, (2) a three-piece structure with a hard intermediate layer between the core and soft cover, and (3) dimple coverage of at least 62%.

2. The '834 Patent

The '834 patent, entitled a "Two-Piece Golf Ball," was applied for at the PTO on February 27, 1997, claiming priority to Japanese patent application no. 8-071135, filed March 1, 1996 . On September 8, 1998, the PTO issued the '834 Patent to Bridgestone. The patent names Hisashi Yamagishi and Jun Shindo as the inventors. This patent generally claims the combination of: (1) a core with a particular hardness profile, whose key features are a hardness gradient of 8-20, and a relatively consistent hardness within the outer 5mm of the core, (2) a two piece structure with a hardness difference between the core and the cover, and a specified cover thickness and, (3) 360 – 450 dimples in the cover.

3. The '791 Patent

On January 20, 2004, over five and a half years after Bridgestone's '707 Patent issued, the PTO issued the '791 Patent, entitled "Golf Ball," to Bridgestone. The only named inventor of the '791 Patent is Hideo Watanabe. The '791 Patent, which like the '707 Patent discloses a three-piece solid golf ball, has claims directed toward the following combination of three elements: (1) a core with a hardness profile which is gradually increasing and a gradient of at least 22 degrees, (2) a three-or-more-piece structure whose key feature is that at least one intermediate layer is harder than the cover and the core, and (3) the use of a compounding agent such as zinc pentachlorothiophenol in the core formulation.

The '791 patent was filed in the PTO on June 15, 2001 and claimed priority to Japanese patent application No. 2000-1960640, filed June 26, 2000.

The original United States application for the '791 Patent claimed a core hardness gradient of "at least 18 degrees," and it only claimed cores with a "gradually increasing" hardness. The core gradient claim was rejected three times by the PTO. In the third office

action, the examiner rejected most of the '791's claims citing Bridgestone's '707 patent as anticipating the '791 claims. In particular, the examiner observed that the '707 patent taught a core with a hardness gradient of 8 to 20. The applicant then amended the claims in response to this office action and replaced the claim of a core gradient of at least 18 with a claim of a gradient of at least 22. The applicant argued to the PTO that, because the '707 patent did not show core gradients over 22, it did not anticipate the amended claim. In response to this, the examiner granted the claims of the '791 patent.

B. Overview of the Technology

1. Hardness Gradients in Rubber Chemistry

I have reviewed the expert report of Dr. Koenig, and I agree with him that formation of hardness gradients in cured rubbers is well-known in rubber chemistry. Scientists and engineers working on rubber molding have known about hardness gradients for decades. Much effort has gone into measuring and modeling the extent of cure in a molded rubber part. Also, academics have written papers on the modeling of the extent of cure in molded rubber parts.[6]

2. Hardness Gradients in the Golf Ball Art

Hardness gradients were known to those in the golf ball field before the earliest priority date of the '707 patent (March 1996). The formation of a hardness gradient in the core is a natural result of the manufacturing process for solid rubber golf balls, and this too was well-known before the Bridgestone patents.

---

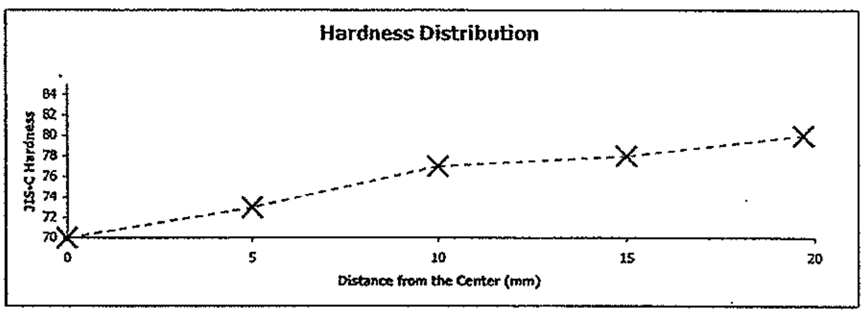
[6] *See* Ex. 28, M.H.R. Ghoreishy & G. Naderi, Three-dimensional Finite Element Modeling of a Rubber Curing Process, 37 J. Elastomers & Plastics 37 (Jan. 2005). In 1980, Prentice and Williams published a paper on modeling the state of cure in a vulcanized rubber article. *See* Ex. 29, G.A. Prentice & M.C. Williams, Numerical Evaluation of the State of Cure in a Vulcanizing Rubber Article, 53 Rubber Chem. Tech. 1023 (1980). In 1990, Kau and Petrusha published a paper on the formation gradients of other physical properties similar to hardness gradients during the molding process. *See* Ex. 30, H.T. Kau & L.A. Pertusha, Dimensional Stability and Property Gradients in Thick Sheet Molding Compound (SMC) Sections, 30 Polymer Engineering & Sci. 805 (July 1990). Lee has published research on calculating the temperature gradient created by the molding process in 1989, as well. *See* Ex. 31, C. Lee, Reaction and Thermal Analysis for SMC (Sheet Molding Compound) Molding in Complicated Geometries, 29 Polymer Engineering & Sci. 1051 (August 1989).

For example, U.S. Patent No. 6,645,496 (Ex. 32), which claimed priority from a 1993 Japanese patent application, shows the hardness distribution of example balls in Table 2 as follows:

TABLE 2

			Comparative Example No.				
			1	2	3	4	5
Core	Formulation	BR-01	100	100	100	100	100
		Zinc diacrylate	30	35	30	38	18
		Zinc Oxide	19	18	19	17.5	27
		Antioxidant	0.5	0.5	0.5	0.5	0.5
		Dicumyl peroxide	1.2	2.0	2.0	1.2	2.2
	Vulcanizing condition		140° C. × 25 min + 165° C. × 8 min	163° C. × 25 min	150° C. × 35 min	160° C. × 30 min	165° C. × 30 min
	Hardness distribution	Center	74	70	69	73	38
		Location which is 5 mm away from the center	75	73	75	76	54
		Location which is 10 mm away from the center	77	77	77	78	58
		Location which is 15 mm away from the center	78	78	80	84	65
		Surface	79	80	82	88	69
	Compression strength	(mm)	2.95	3.05	2.90	2.80	5.00
Cover	Formulation	Hi-milane 1605	50	50	50	50	50
		Hi-milane 1706	50	50	50	50	50
	Stiffness	Kgf/cm^2	3000	3000	3000	3000	3000
	Cover thickness	mm	1.6	1.6	1.6	1.6	1.6

Table 2 shows the hardness at several points between the center and the surface of the core.



Other examples of patents that disclose the presence of a core gradient in a golf ball prior to 1996 include U.S. Patent No. 4,714,253 (Ex. 33) (1987, showing a difference in hardness between the core center and a point 5 to 10 mm from the core center); U.S. Patent No. 5,002,281 (Ex. 34) (1991, showing a hardness at the core center and a point 5 to 10 mm from the core center); U.S. Patent No. 5,184,828 (Ex. 35) (1993, showing hardness in 5mm increments between the core

center and surface); U.S. Patent No. 5,711,723 (Ex. 36) (1995 application, showing a hardness gradient of not more than 4); and U.S. Patent No. 5,730,663 (Ex. 37) (1995 application, showing the hardness in 5mm increments between the core center and surface).

Still other patents show a gradient of greater than 22 degrees before the earliest priority date of the'791 patent (2000). Bridgestone's own U.S. Patent No. 5,830,085 (Ex. 38), for example, showed the use of a gradient of 5 to 25 degrees in a three-piece golf ball in 1998. United States Patent No. 6,390,935 (Ex. 39), filed in 1999, taught a gradient of 8 to 25 degrees. And U.S. Patent No. 6,386,993 (Ex. 40), also filed in 1999, claimed a gradient of 20 to 40 degrees.

3. Hardness Gradient in the Core Manufacturing Process

Solid golf ball cores are manufactured through a curing process. During the curing of a golf ball core, raw polybutadiene is mixed with, among other things, a crosslinking agent or catalyst and heated in a mold. The use of peroxide catalysts, such as dicumyl peroxide, is common in the golf ball art. As the mold is heated, the peroxide breaks down, forming free radicals, which then cause the strands of polybutadiene to form bonds, or "cross-link," with each other. As the temperature increases and the curing time increases, the crosslinking increases. Crosslinking increases the hardness of rubber.

During the core manufacturing process, the rubber is heated by conduction from the hot metal mold. Heat is transferred from the mold to the surface of the cores, and then is conducted inwards. Because polybutadiene has a low thermal conductivity, the surface of the core heats up appreciably faster than the center. Therefore, there is a pronounced temperature difference in the core, with the outside of the core initially being much hotter than the center.

Conventional core molding processes are usually stopped before the core reaches complete thermal equilibrium. Consequently, the outer portions are heated to temperatures where peroxide decomposition and rubber crosslinking occurs for a longer time than the inner

portions. As a result, greater crosslinking occurs at the outside of the core than at the inside. Thus, the outside of the core becomes harder than the center.

Core hardness gradients become larger as the core curing times are decreased, where other influencing factors remain constant. Most golf ball manufacturers try to produce as many balls as they can in a given amount of time. Therefore, there is a tendency in the industry to use the minimum amount of cure time which results in a good product. This results in balls which have hardness gradients.

4. Measuring the Core Hardness Gradient

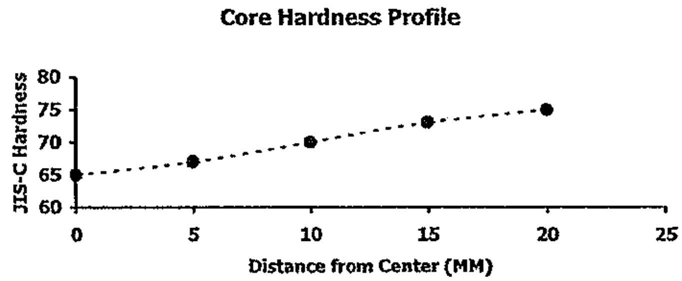
As already discussed, core hardness gradient is a measurement of how the hardness of the core rubber changes from the center of the core to the surface of the core.

Those in the golf ball art use an instrument called a durometer with a specially-shaped indenter to measure "hardness." The indenter is pressed against the core's surface with a defined force, and the durometer measures the amount of deflection of the surface in response to the force. This method of testing core hardness is described in various standards and specifies unique indenters and forces. JIS-C hardness refers to a standard published by the Japan Industry Standard. (*See* Ex. 41.) Shore-D hardness refers to a standard published by ASTM, International. (*See* Ex. 42.) The application of these tests to golf ball components is well-known in the golf ball art. It is understood that these tests are only repeatable to within several degrees, as the standard deviation within a laboratory is known to be roughly 0.8 degrees Shore D, and the reproducibility for tests conducted at different laboratories is roughly 16%.[7] Consequentially, a variation of several degrees between repeated tests of the same specimen is to be expected.

The core surface hardness is taken by first removing the golf ball's cover and intermediate layer, and placing the durometer directly on the core surface. In order to measure hardness at the center of the core, the core is first cut in half. *See* '707 Patent, Col. 7, ll. 8-12.

[7] *See* Ex. 42, ASTM D-2240, Table 4

The durometer is then applied to the center of the core. The hardness can be measured at other points between the center and the surface, as well. If the hardness is taken at many points between the center and the surface, it can be plotted, showing a hardness profile, as follows:



VIII. UNITED STATES PATENT NO. 5,782,707

The '707 patent discloses a three-piece solid golf ball comprising a solid core, an intermediate layer and a cover. Claim 1, the only asserted claim, reads:

> A three piece solid golf ball of the three layer structure comprising a solid core, an intermediate layer and a cover having a plurality of dimples in the ball surface wherein the solid core, intermediate layer, and cover each have a hardness as measured by a JIS-C scale hardness meter wherein the core center hardness is up to 75 degrees, the core surface hardness is up to 85 degrees, the core surface hardness is higher than the core center hardness by 8 to 20 degrees[8], the intermediate layer hardness is higher than the core surface hardness by at least 5 degrees, and the cover hardness is lower than the intermediate layer hardness by at least 5 degrees, and the dimples occupy at least 62% of the ball surface.

('707 Pat., Col. 10, ll. 55-67).

A. The '707 patent is anticipated by Bridgestone's 1994 Altus Newing Massy Golf Ball

I have concluded that claim 1 is invalid as anticipated by Bridgestone's own Altus Newing Massy golf ball. It is my understanding that Bridgestone sold the Altus Newing Massy in Japan since at least the fall of 1994 and that the Altus Newing Massy ball has been known and

[8] A degree is a unit of measurement on the JIS-C scale. JIS-C hardness is measured on a scale of zero to one hundred degrees.

used by Acushnet employees in the United States since November 1994. *See* AB 0004586-88. Therefore, I have treated the Altus Newing Massy as prior art under 35 U.S.C. § 102.

I created a protocol for testing of prior art golf balls. Following my direction, Acushnet engineers tested six 1994 Altus Newing Massy golf balls in accordance this protocol on equipment that I personally inspected to perform the tests. The tests performed were done properly and objectively, and in accordance with the hardness testing prescribed in the '707 patent.[9] I have reproduced the raw data obtained by these tests below, as well as a table containing my analysis of this data.

1994 ALTUS NEWING MASSY MEASURED VALUES[10]

Sample No.	1	2	4	5	6
Cover Hardness (JIS-C)	80.8	80.8	81.3	81.1	79.8
	82.3	81.8	80.7	81.3	81.1
Intermediate Layer Hardness (JIS-C)	97.4	97	97.1	97.4	97.1
	97.3	96.5	97.5	96.8	97.2
Core Surface Hardness (JIS-C)	75.5	78	77.2	75.9	77.2
	76.6	77	77.5	76.3	77.6
Core Center Hardness (JIS-C)	66.1	66.1	64.6	66	63.2

1994 ALTUS NEWING MASSY COMPARISON TO '707 PATENT

Sample No.	1	2	4	5	6

[9] One of the six sample balls, sample number three, is not listed above. Sample three had a core hardness gradient of 5.75 degrees. This is not significant as some golf balls in the same lot may vary from the others. I would expect to see variation in the hardness gradients in a sampling of Altus Newing balls. This does not affect my opinion that the '707 is invalid based on the Altus Newing Massy, as all the other sample balls met each element of claim 1.

[10] This table shows the raw test data. The results of two cover and intermediate layer hardness measurements, measured once at each pole, are shown here. The results of two core surface hardness measurements, again taken once at each pole, are also shown here. Core center hardness was measured once, at the core center. When more than one measurement was taken, the average values are used in the calculations below.

wherein the core center hardness is up to 75 degrees,	66.1	66.1	64.6	66	63.2
the core surface hardness is up to 85 degrees,	76.05	77.5	77.35	76.1	77.4
the core surface hardness is higher than the core center hardness by 8 to 20 degrees	9.95	11.4	12.75	10.1	14.2
The intermediate layer hardness is higher than the core surface hardness by at least 5 degrees,	21.3	19.25	19.95	21	19.75
and the cover hardness is lower than the intermediate layer hardness by at least 5 degrees,	15.8	15.45	16.3	15.9	16.7

My opinion, based on the results of these tests, is that the Altus Newing Massy golf ball includes each and every limitation of claim 1 of the '707 patent, as follows:

- All five balls had a core center hardness between 63 and 67.
- The average core surface hardness, measured at several points on the surface, fell between 76 and 78 degrees.
- The core surface hardness was always higher than the core center hardness by 8 to 20 degrees.
- The intermediate layer hardness, measured at two points on the surface of the sphere, was at least nineteen degrees harder than the core surface.
- The cover hardness, measured on the ball in two locations, was lower than the intermediate layer by at least fifteen degrees.
- Dimple coverage was 76.3%.

It is my opinion that these tests accurately reflect the properties of the golf balls when they were sold. Although the balls are over twelve years old, their multi-layer construction protects the core rubber from the elements, thereby preventing material degradation. It is known in the golf ball art that a polybutadiene rubber core can be adversely affected by moisture and possibly oxygen. But the Altus Newing Massy is encased in an ionomer resin cover and intermediate layer,[11] which would protect the core from the negative affects of moisture and oxygen permeation. In fact, golf ball cores are almost always covered with a material such as an ionomer, particularly the harder ionomers, for the very reason that this provides protection against the penetration of water vapor, thus keeping the core properties from degrading with time.[12] Therefore, many golf balls today have a polyurethane cover and an ionomer intermediate layer; and, as it is known in the art, one of the important functions of the ionomer intermediate layer is to protect the core from moisture penetrations.[13] While a suspected mechanism of core degradation may also be oxidation that can occur, this phenomenon has also been studied, and it has been noted in the art that ionomers act as an oxygen barrier in addition to a water barrier.[14]

My conclusion in this regard is supported by prior competitive testing performed on the Altus Newing Massy ball at Acushnet. AB 0004586-88, for example, shows prior testing of balls acquired by Acushnet in 1994, which indicates that it had a core surface hardness of 77 degrees Shore C. AB 0004583-85 also shows testing of core hardness on balls acquired in 1994, which had a surface hardness of 75 degrees Shore C. The average core surface hardness reported in the table above is 76.9 degrees, which is almost the same.

---

[11] *See* AB 0004587.

[12] Ex. 44, U.S. Patent Application No. 2006/0128505, "Golf ball layers having improved barrier properties."

[13] Ex. 45, Plastics Design Library, "Permeability and Other Film Properties of Plastics and Elastomers."

[14] Ex. 46, U.S. Patent No. 6,398,668, col 1, lines 66-67.

B. The '707 Patent is Obvious in Light of European Patent 0 633 043

It is also my opinion that claim 1 of the '707 patent is invalid based on obviousness in light of the combination of European Patent 0 633 043, which discloses the claimed intermediate layer and core, and the knowledge of one skilled in the art.

EP 0 633 043 ("EP 043") (Ex. 47) issued on April 6, 1997 to Bridgestone, naming Hiroshi Higuchi, Hisashi Yamagishi, and Yoshinori Egashira as inventors of this patent. Mr. Yamagishi and Mr. Higuchi are also the two inventors on the '707 Patent. The EP '043 claims priority to a Japanese application filed in August 1993. As EP '043 claims priority before the priority date of the '707 patent (March 1996), I understand that EP '043 is prior art under 35 U.S.C. § 102(a).

EP '043 claims a three-piece solid golf ball that has a solid core, an intermediate layer and an outer cover layer, just like the golf ball disclosed in the '707 patent. The ball has an intermediate layer which is hard relative to the cover and the core. *See* [0010]. The purpose of the invention is to provide good flight performance, control, feel, and durability. *See* [0009].

The EP 043 patent teaches a core which is formed from a "well known rubber composition." *See* [0017]. Just like the '707 patent, the EP 043 patent provides the core recipe, core diameter, curing time, and curing temperature. *See* Table 1; [0023]. The specification discusses a core hardness of 45 to 80 degrees. *See* [0011].

The EP 043 reference provides nine example balls, and provides detailed instruction as to their manufacture. These instructions include the core composition, curing time, and curing temperature. Table 2 of the EP 043 patent discloses the resulting measurements for the cover hardness, intermediate layer hardness, and core surface hardness of the example balls... Although the inventors did not disclose the core center hardness of the example balls, based upon the teachings of the specification, one of ordinary skill in the art can easily determine that value.

The engineers followed the directions in the EP '043 patent to make and measure the properties of the core. Because the EP '043 patent's instructions are just as detailed as those of

the '707 patent, one of ordinary skill should get a consistent core center hardness by following the instructions in the patent.[15] The engineers manufactured a core and measured the core center hardness at 50.2 degrees. The core had a surface hardness of 67.4 degrees, which is equivalent to the example hardness of 66 degrees in light of the repeatability of durometer measurements.[16]

a. "wherein the core center hardness is up to 75 degrees"

The center hardness of the core manufactured in accordance with the recipe and curing conditions disclosed in EP '043 was 50.2 degrees, which meets this limitation.

b. "the core surface hardness is up to 85 degrees"

In Example 2 of Table 2, the EP '043 patent discloses a core with a surface hardness of 66 degrees, which meets this limitation.

c. "the core surface hardness us higher than the core hardness by 8 to 20 degrees"

The hardness gradient of the core manufactured in accordance with the recipe and curing conditions disclosed in EP '043 was 17.2 degrees, which meets this limitation.

d. "The intermediate layer hardness is higher than the core surface hardness by at least 5 degrees"

Example 2 of Table 2 discloses a core with a surface hardness of 66 degrees and an intermediate layer hardness of 91 degrees. This yields a difference of 25 degrees, which is well within the claimed range.

---

[15] That is, the variation in the results should be consistent with the measurement variation which I discussed above. In addition, since Acushnet owns English-unit molds, the tests used a 35.28 mm core mold in lieu of the 35.31 mm diameter specified in the patent. The difference between these diameters on core hardness is miniscule (0.09%).

[16] The relative repeatability of the Shore-D durometer measurement is 15.7%. *See* ASTM D-2240 (Ex. 42). The JIS-C test has a similar repeatability.

e. "the cover hardness is lower than the intermediate layer hardness by at least 5 degrees"

Table 2 shows that Example 2 has a cover hardness of 82 degrees. *See* Table 2. This is nine degrees lower than the intermediate layer hardness, and therefore meets this limitation.

f. "the dimples occupy at least 62% of the ball surface"

It would have been obvious to one of ordinary skill in the art in March of 1996 to use a dimple pattern of at least 62% when constructing Example 2 in Table 2. The EP '043 does not disclose the dimple coverage of the Example 2 ball. However, it evaluated the flying performance of the example balls, *see* [0027], and example 2 demonstrated good flying performance. *See* Table 2. One of ordinary skill in the golf ball art would recognize that, for a ball to have good flying performance, it would have to have dimples. Therefore, one of ordinary skill in the golf ball are would have looked to what was common in the golf ball art at the time the patentee filed the application that resulted in the EP '043 patent and use a comparable design.

Exhibit 48 is a summary of the percentage dimple coverage for a variety of golf balls from 1992 through 1994, and was generated from Acushnet's competitive test data. During that time period, I understand that Acushnet personnel routinely measured dimple characteristics of competitive golf balls using a profilometer device. Exhibit 48 lists percentage dimple coverage of numerous competitively tested balls, which have been calculated using the phantom sphere method described in the '707 specification. *See* Col. 5, l. 35 – col. 6, l. 15.

As Exhibit 48 shows, almost all of the competitive balls considered between 1992 and 1994 had dimple surface coverages well in excess of 62%. Dimple surface coverage has increased over time – so it would have been obvious to use at least surface coverage as was common at that time – not less. Consequentially, it would have been obvious to one of ordinary skill in the art to use a dimple pattern with at least 62% dimple coverage.

inner cover.[24] A cover type "b" inner cover is a blend of 50 parts Himilan 1706 and 50 parts Himilan 1605. This particular blend of Himilan 1706 and 1605 is common in the prior art, and its properties are well-known. Himilan is an ionomer resin. Therefore, just like the '791 golf ball, the '563 ball has an intermediate layer made of resin.

Also, Bridgestone's '852 patent, published in 1996, shows that this Himilan blend has a JIS-C hardness of 91 degrees. One of ordinary skill in the art knows that it is possible to convert Shore D hardness measurements to the JIC-S scale using a conversion formula. The formula is an approximation based upon experimental tests. DuPont has published one such formula (*See* Ex. 8):

Shore D = (0.76 • JIS C) - 8[25]

U.S. Patent No. 5,645,496, issued in 1997, shows that this Himilan blend has a Shore D hardness of 62. A Shore D hardness of 62 corresponds to a JIS-C hardness of 92 degrees. Bridgestone's U.S. patent No. 6,267,694, shows that the blend has a Shore D hardness of 62 degrees. Therefore, one of ordinary skill in the art would understand that a blend of Himilan 1706 and 1605 has a JIS-C hardness of 91-92 degrees and a Shore D hardness of 62.

As the intermediate layer, with a Shore D hardness of 62 degrees, is harder than the cover, the '563 also meets this limitation of claim 1 of the '791 patent.

Although the '563 does not disclose the hardness of the cores, the '563 patent gives the recipe for each example core. *See* Col 7, l. 45- Col. 8, l. 10. Table 1. The curing time (18 minutes) and temperature (160°) is also provided in the specification. *See* Col, 6, l. 50. Therefore, I had the cores of Example 4 of the '563 patent reproduced in accordance with the

---

[24] One of ordinary skill in the art would understand that the "inner cover" of the '563 patent is the same feature identified as the "intermediate layer" in the '791 patent. Both patents disclose a multi-piece solid golf ball. The Figure 1 in both patents are almost identical and the brief description of the drawings in the '563 and '791 patent identify the same middle layer when referring to the "inner layer" and "intermediate layer," respectively.

[25] I recognize that other conversion formulas are also used in the golf ball art. Bridgestone's own documents list three other conversion formulae: (1) Shore D = (0.886 • JIS C) – 16.31; (2) Shore D = (0.7637 • JIS C) – 8.2925; and (3) Shore D = (0.7982 • JIS C) – 10.28. (*See* Ex. 51.)

XI. CONCLUSION

I reserve the right to supplement this report should new information come to light that bears on my opinions contained in this report. I reserve the right to supplement or modify this report, if appropriate, to the extent that new or additional information is provided. I also reserve the right to consider and comment on additional evidence that may be presented by experts for Bridgestone.

At trial or any hearing in this litigation, I may provide demonstrative aids, such as computer animations, excerpts from relevant exhibits, deposition testimony, and physical examples, to assist in explaining the subject matter discussed in this report.

Signed this sixteenth day of January, 2007.

David Felker, Ph.D.

TAB 1

David L. Felker, PhD
8852 Harmony Grove Road
Escondido, CA 92029
760-468-0816(mobile)
760-480-7515 x13(office)

2/01-present Owner, Sanford Rose Associates- Escondido

- Retained executive search firm filling primarily Director to CEO positions in the Pharmaceutical and Biotechnology industries throughout the USA.

1/01 – present Golf Ball Industry Consultant/Expert Witness

- Provide technical advice and expert testimony in golf ball technology-relayed cases.
- Perform patent analysis and provide technical advice.
- Lead scientific efforts to demonstrate performance differences between golf products for the purpose of providing evidence to support the client's legal position.
- Perform/direct physical property testing and outdoor performance comparison testing efforts.
- Direct/perform statistically designed experiments and thorough analysis of data.

Depositions:

5/8/01 Callaway Golf v. Bridgestone Sports Co. Ltd., Civil Action No. 1 00-CV-1871-JEC

3/6/02 Callaway Golf Company v. Dunlop Slazenger Group Americas, Inc.
Civil Action No. 01-CV-669 RRM

5/22/02 Callaway Golf Company v. Dunlop Slazenger Group Americas, Inc.
Civil Action No. 01-CV-669 RRM

2003 Nitro Leisure Products, LLC v. Acushnet Company, US District Court Southern District of Florida, Case No. 02-14008-CIV-Middlebrooks

Declarations:

Callaway Golf v. Acushnet Company, 7/00

Callaway Golf v. Bridgestone Sports Co. Ltd., 8/00

Acushnet Company v. Nitro Leisure Products, LLC dba Golf ballsdirect.com and Second Chance, and Nitro Leisure Services, LLC dba Nitro Golf and Nitrogolf.com, US District Court Southern District of Florida, Case No. 02-14091-CIV-Roettger, 4/19/02

Nitro Leisure Products, LLC v. Acushnet Company, US District Court Southern District of Florida, Case No. 02-14008-CIV-Middlebrooks, 6/11/02

Nitro Leisure Products, LLC v. Acushnet Company, US District Court Southern District of Florida, Case No. 02-14008-CIV-Middlebrooks, 8/6/02

Nitro Leisure Products, LLC v. Acushnet Company, US District Court Southern District of Florida, Case No. 02-14008-CIV-Middlebrooks, 5/28/03

Expert Reports:
"Remanufactured Golf Ball Testing and Results", 4/18/02

Expert Report for Nitro Leisure Products, LLC v. Acushnet Company, US District Court Southern District of Florida, Case No. 02-14008-CIV-Middlebrooks, 7/8/03

Courtroom Testimony:
8/04 Callaway Golf Company v. Dunlop Slazenger Group Americas, Inc.
Civil Action No. 01-CV-669 RRM

12/96-10/00 Vice President of Research & Development, Callaway Golf Ball Company, Carlsbad, CA (Start-Up Company wholly owned subsidiary of Callaway Golf Company)

- Charter member of executive team that built $170 M Start-up Company from scratch.
- Designed and developed the entire R&D function and company's first products; $6M R&D annual operating budget; jointly responsible for Golf Ball Company P&L.
- Lead the effort that produced four *Demonstrably Superior and Pleasingly Different* (DSPD) golf ball models: *Rule 35, CTU 30, CB-1* and *HX*". Total sales for these four products were $55M in 2001. The Rule 35's performance set a new standard that stunned the golf ball industry. Additionally, the "HX" is revolutionary in that it is a "dimple free" golf ball.
- Lead the patent effort that enabled the introduction of patented products in an extremely crowed art; did so without legal issues at product launch.
- Inventor of golf ball/club products and golf ball manufacturing processes.

  PATENTS
  - 5,984,807 Golf Ball
  - 6,200,512 Method of Manufacturing a Golf Ball
  - 6,213,892 Multi-layer Golf Ball
  - 6,245,386 Method and system for finishing a golf ball
  - 6,390,932 Compliant polymer face golf club head
  - 6,440,346 Method for making golf ball
  - 6,607,451 Compliant polymer face golf club head
  - 6,786,837 Golf balls and methods of manufacturing the same

11/94-12/96: Technology Superintendent – Neoprene, DuPont Dow Elastomers, Louisville, KY

- Responsible for: World-wide Neoprene Adhesives and Latex Development & Technical Services, Hypalon™ Adhesives Development & Tech Services, Louisville Manufacturing Technical Support (worlds largest polychloroprene facility), Capital Project Implementation ($15-25M in capital projects/yr), World-wide Neoprene Research & Development, Technical support of Quality Control and Environmental Laboratories
- Directed 50 person organization; $6M Technology Budget
- Leader of high priority Task Team of scientists from DuPont, DuPont-Dow Elastomers and various Universities working on the next generation process and products.

9/91-11/94: Technical Area Superintendent - Neoprene, DuPont Company, Louisville, KY

- Responsible for: Manufacturing Engineering & Process Support, Capital Project Implementation ($10-40M in Capital projects/yr), ISO 9000 Certification, Quality Control and Environmental Laboratories, and Technical Computing Upgrade. Managed 16 person organization; $2M Technology Budget.
- Working-Leader of International Task Team that solved 40 yr old Neoprene Quality/Manufacturing problem, delivering >$10M/yr ATOI.

9/90-9/91: Hypalon™ Technology Area Superintendent
Elastomers Division, DuPont Beaumont Works, Beaumont, TX

Responsibilities same as 9/89-9/90 position plus responsible for :

- Research & Development of a CCl_4-free Commercial HYPALON™ Process.
- Process development and production of chlorinated EVA/PE polymer in elastomer plant.
- Montreal Protocol liaison for HYPALON™ Business.
- Assembled and lead task team of scientists, lobbyists, and lawyers who developed and implemented a creative win/win solution to a HYPALON™ environmental problem that literally saved the business ($>30M NPV). Received DuPont Board of Directors Award for preventing Shutdown of HYPALON™ Business.

9/89-9/90: Hypalon™ Manufacturing Technology Area Superintendent

- Managed group of 10 responsible for Plant Manufacturing Engineering, Capital Project Implementation, Technical support of Quality Control and Environmental Laboratories
- Received DuPont Award for increasing plant production of Cl-EVA 400% using fundamental process understandings and statistically designed experiments.

9/86-9/87: Product Development Engineer: Nylon Compounding/Elastomer Toughened Polymers, DuPont Washington Works Plant, Parkersburg, WV

Developed family of elastomer toughened/reinforced ZYTEL™ compounded products with super high flow properties. Responsible for plant engineering support for carbon black filled Nylon and polyester products.

9/87-9/89 Manufacturing Engineer: DuPont Advanced Glazing Venture, Parkersburg, WV

Part of Team that successfully developed and introduced the Anti-Lacerative Windshield™ and SpallShield™. Primarily responsible for pilot plant operation and managing contract coating operations with w/ Polaroid Corp, Custom Coating & Laminating Inc. and Rexham Corp.

10/84-9/86: Research Engineer Long Range Research Group
Polymer Products Department, DuPont Experimental Station, Wilmington, DE

Product and Process R&D focused on a ARYLON™ (new aromatic polyester targeted at the auto industry).

EDUCATION:
Aug 1984: Iowa State University, Ames, Iowa
Ph.D. Chemical Engineering. Thesis: Electrochemical dissolution of copper sulphide minerals using a Fluidized Bed Electrochemical Reactor.
Recipient of the Iowa State University Mining & Mineral Resources Research Institute Fellowship, 1981-1983. Overall G.P.A. 3.69/4.00

M.S. Chemical Engineering Aug 1982. Thesis work included experimental and mathematical studies dealing with the electrodeposition of copper using a fluidized bed electrochemical reactor. Overall G.P.A. 3.68/4.00

University of Wisconsin-Eau Claire, Eau Claire, Wisconsin 1976-1979
Received B.S. in Chemistry May of 1979. Class work included advanced organic chemistry and several semesters of independent research. Overall G.P.A. 3.20/4.00

OTHER ACADEMIC RESEARCH EXPERIENCE:
1979--National Science Foundation Undergraduate Research Participant. Worked under the guidance of Dr. George A. Kraus, Department of Chemistry, Iowa State University. Research included the investigation of the reaction of dienolate anions with Michael acceptors.

1977-1979--Research Assistant at the University of Wisconsin-Eau Claire. Worked under the guidance of Dr. William C. Groutas. Research included the synthesis of functionalized alpha methylene valerolactones fused to substituted aromatic rings, pancreatic elastase inhibiting imidazole-N-carboxamides, and N,N'-ethylene bis [2(2-hydroxy-5bromophenyl)l glycine.

PAPERS PUBLISHED
William C. Groutas and David L. Felker, "Synthetic applications of Cyanotrimethylsilane, Iodotrimethylsilane, Azidotrimethylsilane, and Methylthiotrimethylsilane," Synthesis, No. 11, 861-868, November (1980).

William C. Groutas, David L. Felker, David R. Magnin, George Meitzner, and Terry Gaynor, "Synthesis of Functionalized Alpha-Methylene Lactones" Synthetic Communications 10 (1), 1-9 (1980)

William C. Groutas, David L. Felker and David R. Magnin, "Synthesis of Aromatic AlphaMethylene Lactones", Synthetic Communications 10 (5), 355-362 (1980).

William C. Groutas, R.C. Badger, T.D. Ocain, D.L. Felker, J. Frankson and M. Theodorakis, "Mechanism-Based Inhibitors of Elastase", Biochemical and Biophysical Research Communications, Vol. 95, No. 4, 1980-1894 (1980).

Michael C. Theodorakis, William C. Groutas, Alex J. Bermudez, David L. Felker, StavroulaVani Stefanakou, David R. Magin, and Terry Gaynor, "Localization of Technetium-99mN,N'-ethylene-bis [2(2-hyd roxy-5-b romophenyl)] glycine and Technetium-99m-[N-2(2mercapto-l-oxo-propylglycine)] in the Hepatobiliary System",
Submitted to J. Pharmaceutical Sciences for publication.

David L. Felker and Renato G. Bautista, "The Electrowinning of Copper Using a Side-By-Side Fluidized Bed Electrochemical Reactor", published in IE&C Process Design and Development.

David L. Felker and Renato G. Bautista, Electrochemical Processes in Recovering Metals from Ores, Journal of Metals, April 1990, 60-63.

Videotape : How to Grow Gourmet Oyster Mushrooms, April 1995

"Advances in Neoprene Technology", 1996 (definitive scientific text internally published in DuPont under my direction)

PAPERS PRESENTED:

22nd Undergraduate Chemistry Symposium, Minnesota Chapter of the American Chemical Society, Bethel College, Spring 1978, "Design and Synthesis of Potential Cytotoxic Agents", coauthored by William C. Groutas and David L. Felker, presented by David L. Felker.

Society of Mining Engineers of the AIME Fall Meeting and Exhibit, Minneapolis Auditorium and Convention Hall, Minneapolis, Minn., Oct. 22-24, 1980, "The Electrowinning of Copper from Sulphate Solutions in a Fluidized Bed Electrochemical Reactor", coauthored by Chang C. Ko, David L. Felker, Harvey Jensen, and Renato G. Bautista, presented by David Felker.

The Metallurgical Society of the AIME Annual Meeting, Dallas Convention Center, Dallas, Texas, Feb. 14-18, 1982, "Design Considerations for the Scale-Up of a Fluidized Bed Electrochemical Reactor (FBER)", coauthored by David L. Felker and Renato G. Bautista, presented by David L. Felker.

The Metallurgical Society of the AIME Annual Meeting, Hyatt Regency Hotel, Atlanta, Georgia, March 6-10, 1983, "A Model for Predicting the Concentration-Time Relationship using a FBER", coauthored by David L. Felker and Renato G. Bautista, presented by Renato G. Bautista.

TMS-SME Annual Mtg, Denver, Colorado, Feb 24-27, 1987 , "A mathematical model for the electrochemical reduction of chalcopyrites using a fluidized bed electrochemical reactor", coauthored by David L. Felker and Renato G. Bautista, presented by David L. Felker.

TMS-SME Annual Mtg, Denver, Colorado, Feb 24-27, 1987 , "The two-stage dissolution and separation of Cu, Fe, and S from chalcopyrite using a fluidized bed electrochemical reactor", coauthored by David L. Felker and Renato G. Bautista, presented by David L. Felker.

Proprietary Presentations at DuPont Polymer Products Department annual TECH-CON, 1985, 1986.

Proprietary Presentation at DuPont Polymer Products Department annual Polymer Compounding Conference, Parkersburg WV, 1987.

PERSONAL DATA:

U.S. Citizen, married, born 6/10/57, 6'1", 200 lbs., excellent health.
Hobbies and interests include swimming, painting, hiking, skiing, tennis, flying military aircraft, golf and fly-fishing.

TAB 41

JIS

JAPANESE INDUSTRIAL STANDARD

Translated and Published by
Japanese Standards Association

JIS K 6253 : 1997

Hardness testing methods for rubber, vulcanized or thermoplastic

ICS 83.060

Descriptors : vulcanized rubber, vulcanized materials, hardness testing, mechanical testing, hardness, mechanical properties of materials

Reference number : JIS K 6253 : 1997 (E)

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law:

Date of Establishment: 1993-02-01

Date of Revision: 1997-04-20

Date of Public Notice in Official Gazette: 1997-04-21

Investigated by: Japanese Industrial Standards Committee
Divisional Council on Chemical

JIS K 6253:1997, First English edition published in 1998-12

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

In the event of any doubts arising as to the contents, the original JIS is to be the final authority.

Printed in Japan

Hardness testing methods for rubber, vulcanized or thermoplastic

Introduction This Japanese Industrial Standard has been prepared on the basis of the 3rd edition of **ISO 48**, *Rubber, vulcanized or thermoplastic—Determination of hardness (hardness between 10 IRHD and 100 IRHD)* published in 1994, and the 1st edition of **ISO 7619**, *Rubber—Determination of indentation hardness by means of pocket hardness meters* published in 1986, without any modification in technical contents. However, "Type E of spring type (durometer hardness)" which is not specified in the corresponding International Standards are added in this Standard.

1 Scope This Japanese Industrial Standard specifies the testing methods to measure hardness of vulcanized rubber and thermoplastic rubber (hereafter referred to as "vulcanized rubber").

Remarks 1 The standards cited in this Standard are listed as follows.

JIS K 6200 *Glossary of terms used in rubber industry*

JIS K 6250 *General rules of physical testing methods for rubber, vulcanized or thermoplastic*

JIS Z 8401 *Rules for rounding off of numerical values*

2 The International Standards corresponding to this Standard are listed as follows.

ISO 48 : 1994 *Rubber, vulcanized or thermoplastic—Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 7619 : 1986 *Rubber—Determination of indentation hardness by means of pocket hardness meters*

3 The units and numerical values given in { } in this Standard are based on traditional units, and are appended for informative reference.

2 Definitions For the purposes of this Standard, the definitions given in **JIS K 6200** and **JIS K 6250**, and the following definitions apply.

(1) **international rubber hardness degree** Hardness which can be obtained through conversion into international rubber hardness degree (IRHD)(1) using the depth of indentation by a plunger when the plunger, with a ball-type lower end, is vertically impressed on the surface of a test piece with specified indenting force.

A hardness scale is chosen so that "0" represents the hardness of material having a Young's modulus of zero and "100" represents the hardness of a material of infinite Young's modulus, and the following conditions are fulfilled over most of normal range of hardness.

(a) One international rubber hardness degree always represents approximately the same proportionate difference in the Young's modulus.

(b) For highly elastic rubber, the scales of international rubber hardness degree and that of type A durometer are comparable.

Note (1) IRHD International Rubber Hardness Degree

(2) **durometer hardness** The hardness given by the testing apparatus (durometer) which reads the indentation depth made by a specifically shaped indentor when it is impressed on the surface of a test piece via a spring.

(3) **IRHD pocket hardness** The hardness given by a portable pocket testing apparatus (IRHD pocket hardness meter) by which international rubber hardness degree can be conveniently obtained owing to reading the indented depth made by an indentor, with a ball-type lower end, when it impressed on the surface of a test piece via a spring.

(4) **standard hardness** The hardness obtained using the specified procedures on test pieces whose shape and dimensions satisfy the specifications, when carrying out each test.

(5) **apparent hardness** The hardness obtained either using other procedures than the specified, or on the test piece whose shape and dimensions do not satisfy the specification, when carrying out each test.

3 Type of test

3.1 Outline of hardness test There are many types of testing methods for hardness test depending on the principle of hardness measurement, range of hardness measurement, kind of testing apparatus and so on, and they are classified into standard hardness and apparent hardness by shape or dimensions of a test piece. The outline of classifying is shown in Table 1.

Table 1 Outline of hardness tests

Principle of measurement	Range of hardness measurement	Type of testing apparatus	Testing method	Test condition for standard hardness		
				Shape	Thickness mm	Minimum distance from the edge of sample mm
Constant-force type (international rubber hardness degree)	For high hardness (85 to 100 IRHD)	Normal size international rubber hardness meter	H method	Both upper and lower surfaces are smooth and parallel each other.	8.0 min.	9.0
					10.0 max.	10.0
	For normal hardness (30 to 95 IRHD)	Normal size international rubber hardness meter	N method		8.0 min.	9.0
					10.0 max.	10.0
		Microsize international rubber hardness meter	M method		1.5 min.	2.0
					2.5 max.	
	For low hardness (10 to 35 IRHD)	Normal size international rubber hardness meter	L method		10.0 min.	10.0
					15.0 max.	11.5
Spring type (durometer hardness)	For high hardness (A90 or more)	Type D durometer			6.0 or more	12.0
	For normal hardness (A10 to 90)	Type A durometer			6.0 or more	12.0
	For low hardness (A20 or less)	Type E durometer			10.0 or more	12.0
Spring type (IRHD pocket hardness)	For normal hardness (30 to 95 IRHD)	IRHD pocket hardness meter	P method		6.0 or more	12.0

3.2 Type of tests The type of hardness tests for vulcanized rubber shall be classified as follows.

(1) **International rubber hardness test**

(a) H method (normal size test for high hardness)

(b) N method (normal size test for normal hardness)

(c) M method (microsize test for normal hardness)

(d) L method (normal size test for low hardness)

(2) **Durometer hardness test**

(a) Type D (test for high hardness)

(b) Type A (test for normal hardness)

(c) Type E (test for low hardness)

(3) **IRHD pocket hardness test**

(a) P method (for normal hardness)

4 International rubber hardness test

4.1 Purpose This test shall be carried out to measure the international rubber hardness degree of vulcanized rubber.

4.2 Range of measurement The measuring range of this test is decided according to the thickness and hardness of a test piece for every testing method. The measuring range of each testing method is as follows.

(1) **H method** Formal measuring range shall be for the test piece measuring 8.0 mm to 10.0 mm in thickness and with hardness of 85 IRHD to 100 IRHD. It is permissible to test the one with 4.0 mm or more thickness and with hardness of 85 IRHD to 100 IRHD.

(2) **N method** Formal measuring range shall be for the test piece measuring 8.0 mm to 10.0 mm in thickness and with hardness of 35 IRHD to 85 IRHD. It is permissible to test the one with 4.0 mm or more thickness and with hardness of 30 IRHD to 95 IRHD(2).

(3) **M method** Formal measuring range shall be for the test piece measuring 1.5 mm to 2.5 mm in thickness and with hardness of 35 IRHD to 85 IRHD. It is permissible to test the one with 1.0 mm to 4.0 mm thickness and with hardness of 30 IRHD to 95 IRHD(3).

(4) **L method** Formal measuring range shall be for the test piece measuring 10.0 mm to 15.0 mm in thickness and with hardness of 10 IRHD to 35 IRHD. It is permissible to test the one with 6.0 mm or more thickness and with hardness of 10 IRHD to 35 IRHD.

Notes (2) The hardness values in 85 IRHD to 95 IRHD and 30 IRHD to 35 IRHD obtained by N method do not exactly coincide with the values by H method and L method, but the discrepancy does not come into technical problem, generally speaking.

(3) The testing apparatus for M method is the one prepared by miniaturizing the testing apparatus for N method by about one-sixth to measure the test piece with thin thickness, therefore the depth of plunger indentation by M method is just one-sixth that by N method. The results given by M method are not always coincident with the results given by N method because of the surface effect of rubber or slight roughness of the surface.

4.3 Testing apparatus

4.3.1 Outline of testing apparatus The testing apparatus is composed of a holding base for test piece by which a test piece is kept, an annular pressure foot by which the surface of a test piece is pressed, a plunger, with a ball-type lower end, set at the center of hole of pressure foot, a device for loading which gives an indenting force on a plunger to make an indentation on a test piece, a measuring device to measure depth of an indentation impressed on a test piece, and a vibrating device to lessen friction. The dimensions of main parts and the specification of force are shown in Table 2.

A thermostat may be provided for measuring a test temperature other than standard condition of laboratory.

Table 2 Main dimensions and forces of testing apparatus

Type of tests	Diameter of ball of plunger end mm	Face of pressure foot			Force applying at ball of plunger end		
		Diameter mm	Diameter of hole mm	Force exerted on face of pressure foot	Contact force	Indenting force	Total
H method	1.00±0.01	20±1	6±1	8.3±1.5 N {846±153 gf}	0.30±0.02 N {30.6±2.0 gf}	5.40±0.01 N {550.6±1.0 gf}	5.70±0.03 N {581.2±3.1 gf}
N method	2.50±0.01	20±1	6±1				
L method	5.00±0.01	22±1	10±1				
M method	0.395±0.005	3.35±0.15	1.00±0.15	(4) 235±30 mN {24.0±3.1 gf}	8.3±0.5 mN {0.85±0.05 gf}	145±0.5 mN {14.79±0.05 gf}	153±1 mN {15.60±0.10 gf}

Note (4) When in M method a pressure adjusting spring installed at the bottom of a test-piece holding base makes pressure adjustment, the pressure adjusting spring must be controlled to be (380±30) mN {(38.7±3.1) gf} because an indenting force 145 mN {14.8 gf} is added during measurement.

4.3.2 Face of pressure foot An annular pressure foot makes rectangular to a plunger. The diameter of face of pressure foot and the diameter of the hole for a plunger are as shown in Table 2. When the force exerted on the face of pressure foot is just as shown in Table 2, the pressure impressed on the surface of test piece becomes (30±5) kPa {(0.306±0.051) kgf/cm²}(5). In order to measure the relative displacement between the face of pressure foot (upper surface of test piece) and the plunger, the face of pressure foot shall be firmly united with the measuring device of the depth of indentation.

Note (5) Some combination of all tolerances shown in Table 2 does not always give nice coincidence with the description of pressure (30±5) kPa {(0.306±0.051) kgf/cm²}.

4.3.3 Plunger The plunger shall be vertical, and its lower end has spherical shape whose diameter shall be as shown in Table 2(6). The lower end ball of a plunger shall be kept a little upper than the face of pressure foot before contact force is applied.

Note (6) The material of end ball shall be abrasion resistant and corrosion resistant.

When an end ball is connected with the body of plunger, the connected part must not be larger than diameter of the ball.

4.3.4 Loading device Loading device shall accurately apply the contact force(7) and indenting force(8) specified in Table 2 to the end ball of a plunger.

Notes (7) Contact force means the force causing the end ball of a plunger to contact with surface of a test piece.

(8) Indenting force means the force to impress the end ball of a plunger into test piece after making contact.

4.3.5 Measuring device of indented depth The measuring device for indented depth shall be capable of measuring indented depth of a plunger when indenting force is applied to a plunger, by which the indented depth or IRHD shall be directly read(9). The conversion from indented depth to IRHD can be done through Table 3, Table 4 and Table 5(10).

Notes (9) For the measuring device of indented depth, any of mechanical, optical, or electrical, is serviceable.

(10) Table 3 is for the conversion of H method, and Table 4 for N method. In case of M method, convert after making the indented depth shown in Table 4 one-sixth. Table 5 is the conversion table for L method.

4.3.6 Vibrating device To overcome minute friction, it is preferable to install a vibrating device like an electric buzzer by which a testing apparatus is suitably vibrated. It can be eliminated if friction is completely removed.

4.3.7 Thermostat The thermostat is needed when the test temperature other than standard condition of laboratory is employed for measuring hardness. The thermostat must keep the specified temperature in the tolerance of ±2 °C. The annular foot with pressure face at lower end and a plunger shall penetrate through the upper part of the thermostat.

The part through which the plunger penetrates shall be made of the material with small thermal conductivity. The sensor for temperature measurement shall be installed at holding place of test piece or its vicinity, in the thermostat.

Table 3 Conversion table from indented depth (D) of a plunger to international rubber hardness degree (IRHD) (H method)

D mm	International rubber hardness degree IRHD	D mm	International rubber hardness degree IRHD	D mm	International rubber hardness degree IRHD
0.00	100.0	0.15	97.3	0.30	91.1
0.01	100.0	0.16	97.0	0.31	90.7
0.02	100.0	0.17	96.6	0.32	90.2
0.03	99.9	0.18	96.2	0.33	89.7
0.04	99.9	0.19	95.8	0.34	89.3
0.05	99.8	0.20	95.4	0.35	88.8
0.06	99.6	0.21	95.0	0.36	88.4
0.07	99.5	0.22	94.6	0.37	87.9
0.08	99.3	0.23	94.2	0.38	87.5
0.09	99.1	0.24	93.8	0.39	87.0
0.10	98.8	0.25	93.4	0.40	86.6
0.11	98.6	0.26	92.9	0.41	86.1
0.12	98.3	0.27	92.5	0.42	85.7
0.13	98.0	0.28	92.0	0.43	85.3
0.14	97.6	0.29	91.6	0.44	84.8

Table 4 Conversion table from indented depth (*D*) of a plunger to international rubber hardness degree (IRHD) (N method)

D mm	International rubber hardness degree IRHD	*D* mm	International rubber hardness degree IRHD	*D* mm	International rubber hardness degree IRHD	*D* mm	International rubber hardness degree IRHD
0.00	100.0	0.45	73.9	0.90	52.3	1.35	38.9
0.01	100.0	0.46	73.3	0.91	52.0	1.36	38.7
0.02	99.9	0.47	72.7	0.92	51.6	1.37	38.4
0.03	99.8	0.48	72.2	0.93	51.2	1.38	38.2
0.04	99.6	0.49	71.6	0.94	50.9	1.39	38.0
0.05	99.3	0.50	71.0	0.95	50.5	1.40	37.8
0.06	99.0	0.51	70.4	0.96	50.2	1.41	37.5
0.07	98.6	0.52	69.8	0.97	49.8	1.42	37.3
0.08	98.1	0.53	69.3	0.98	49.5	1.43	37.1
0.09	97.7	0.54	68.7	0.99	49.1	1.44	36.9
0.10	97.1	0.55	68.2	1.00	48.8	1.45	36.7
0.11	96.5	0.56	67.6	1.01	48.5	1.46	36.5
0.12	95.9	0.57	67.1	1.02	48.1	1.47	36.2
0.13	95.3	0.58	66.6	1.03	47.8	1.48	36.0
0.14	94.7	0.59	66.0	1.04	47.5	1.49	35.8
0.15	94.0	0.60	65.5	1.05	47.1	1.50	35.6
0.16	93.4	0.61	65.0	1.06	46.8	1.51	35.4
0.17	92.7	0.62	64.5	1.07	46.5	1.52	35.2
0.18	92.0	0.63	64.0	1.08	46.2	1.53	35.0
0.19	91.3	0.64	63.5	1.09	45.9	1.54	34.8
0.20	90.6	0.65	63.0	1.10	45.6	1.55	34.6
0.21	89.8	0.66	62.5	1.11	45.3	1.56	34.4
0.22	89.2	0.67	62.0	1.12	45.0	1.57	34.2
0.23	88.5	0.68	61.5	1.13	44.7	1.58	34.0
0.24	87.8	0.69	61.1	1.14	44.4	1.59	33.8
0.25	87.1	0.70	60.6	1.15	44.1	1.60	33.6
0.26	86.4	0.71	60.1	1.16	43.8	1.61	33.4
0.27	85.7	0.72	59.7	1.17	43.5	1.62	33.2
0.28	85.0	0.73	59.2	1.18	43.3	1.63	33.0
0.29	84.3	0.74	58.8	1.19	43.0	1.64	32.8
0.30	83.6	0.75	58.3	1.20	42.7	1.65	32.6
0.31	82.9	0.76	57.9	1.21	42.5	1.66	32.4
0.32	82.2	0.77	57.5	1.22	42.2	1.67	32.3
0.33	81.5	0.78	57.0	1.23	41.9	1.68	32.1
0.34	80.9	0.79	56.6	1.24	41.7	1.69	31.9
0.35	80.2	0.80	56.2	1.25	41.4	1.70	31.7
0.36	79.5	0.81	55.8	1.26	41.1	1.71	31.6
0.37	78.9	0.82	55.4	1.27	40.9	1.72	31.4
0.38	78.2	0.83	55.0	1.28	40.6	1.73	31.2
0.39	77.6	0.84	54.6	1.29	40.4	1.74	31.1
0.40	77.0	0.85	54.2	1.30	40.1	1.75	30.9
0.41	76.4	0.86	53.8	1.31	39.9	1.76	30.7
0.42	75.8	0.87	53.4	1.32	39.6	1.77	30.5
0.43	75.2	0.88	53.0	1.33	39.4	1.78	30.4
0.44	74.5	0.89	52.7	1.34	39.1	1.79	30.2
						1.80	30.0

Table 5 Conversion table from indented depth (*D*) of a plunger to international rubber hardness degree (IRHD) (L method)

D mm	International rubber hardness degree IRHD	*D* mm	International rubber hardness degree IRHD	*D* mm	International rubber hardness degree IRHD
1.10	34.9	1.80	21.3	2.50	14.1
1.12	34.4	1.82	21.1	2.52	14.0
1.14	33.9	1.84	20.8	2.54	13.8
1.16	33.4	1.86	20.6	2.56	13.7
1.18	32.9	1.88	20.3	2.58	13.5
1.20	32.4	1.90	20.1	2.60	13.4
1.22	31.9	1.92	19.8	2.62	13.3
1.24	31.4	1.94	19.6	2.64	13.1
1.26	30.9	1.96	19.4	2.66	13.0
1.28	30.4	1.98	19.2	2.68	12.8
1.30	30.0	2.00	18.9	2.70	12.7
1.32	29.6	2.02	18.7	2.72	12.6
1.34	29.2	2.04	18.5	2.74	12.5
1.36	28.8	2.06	18.3	2.76	12.3
1.38	28.4	2.08	18.0	2.78	12.2
1.40	28.0	2.10	17.8	2.80	12.1
1.42	27.6	2.12	17.6	2.82	12.0
1.44	27.2	2.14	17.4	2.84	11.8
1.46	26.8	2.16	17.2	2.86	11.7
1.48	26.4	2.18	17.0	2.88	11.6
1.50	26.1	2.20	16.8	2.90	11.5
1.52	25.7	2.22	16.6	2.92	11.4
1.54	25.4	2.24	16.4	2.94	11.3
1.56	25.0	2.26	16.2	2.96	11.2
1.58	24.7	2.28	16.0	2.98	11.1
1.60	24.4	2.30	15.8	3.00	11.0
1.62	24.1	2.32	15.6	3.02	10.9
1.64	23.8	2.34	15.4	3.04	10.8
1.66	23.5	2.36	15.3	3.06	10.6
1.68	23.1	2.38	15.1	3.08	10.5
1.70	22.8	2.40	14.9	3.10	10.4
1.72	22.5	2.42	14.8	3.12	10.3
1.74	22.2	2.44	14.6	3.14	10.2
1.76	21.9	2.46	14.4	3.16	10.1
1.78	21.6	2.48	14.3	3.18	9.9

4.4 Test piece

4.4.1 Shape of test pieces Both surfaces of a test piece shall be smoothly flat and parallel each other(11). This test has been supposed to compare the test pieces having the same thickness.

Note (11) The surface such as unsmoothed, curved, or rough, does not give satisfactory results. For specially formed surface, however, such as rubber roll, this method can be applied.

The international rubber hardness testing method for curved test piece is shown in Informative reference.

4.4.2 Thickness

(1) **H method and N method** The standard thickness of a test piece is 8.0 mm to 10.0 mm, but to get necessary thickness, it is permissible to pile smooth and parallel test pieces. Provided that the thickness of test pieces before piling shall be 2 mm or more, and 3 or more test pieces cannot be piled up. Even when nonstandard test piece other than above(12) is to be adopted, the thickness of the test piece must be 4.0 mm or more.

(2) **L method** The standard thickness of a test piece is 10.0 mm to 15.0 mm, but to get necessary thickness, it is permissible to pile smooth and parallel test pieces. Provided that the thickness of test pieces before piling shall be 2 mm or more, and 3 or more test pieces cannot be piled up. Even when nonstandard test piece other than above(12) is to be adopted, the thickness of the test piece must be 6.0 mm or more.

(3) **M method** The standard thickness of a test piece is (2.0 ±0.5) mm. Even when nonstandard test piece other than above(12) is to be adopted, the thickness of the test piece must be 1.0 mm or more.

Note (12) The measured value resulted from nonstandard test piece, is not generally coincident with the measured value by standard test piece.

4.4.3 Lateral dimensions

(1) **H method, N method, and L method** The lateral dimension of a test piece shall be large enough to measure at the point which is apart from edge of the test piece by at least the distance shown in Table 6.

Table 6 Minimum distance of point for hardness measurement (point of end ball of plunger) from test-piece edge

Unit: mm

Thickness of a test piece	Minimum distance of point for hardness measurement from test-piece edge
4.0	7.0
6.0	8.0
8.0	9.0
10.0	10.0
15.0	11.5
25.0	13.0

(2) **M method** The lateral dimension of a test piece shall be large enough to measure at the point which is apart from edge of the test piece by at least 2.0 mm. When the test piece, with the thickness of 4.0 mm or more, which is not eligible for N method because of small lateral dimension or of not having large smooth area, is to be tested by M method, carry out test at the point apart from edge of the test piece as far as possible.

4.4.4 Sampling and preparation of test pieces The sampling and preparation of test pieces shall principally follow **6.5** of **JIS K 6250**.

4.4.5 Selection of test pieces The test pieces which contain alien matters, bubbles, or flaws shall not be used for tests.

4.5 Testing method

4.5.1 Testing conditions Testing conditions shall be as follows.

(1) The standard conditions of a laboratory shall follow **6.1** of **JIS K 6250**.

(2) Storing of sample and test pieces shall follow **6.2** of **JIS K 6250**.

(3) The standard conditions of test pieces shall follow **6.3** of **JIS K 6250**.

4.5.2 Procedures Sprinkle slightly talc on upper and back surfaces of a test piece to lessen friction between the end ball of a plunger and surface of a test piece. Place the test piece on the holding base of a test piece. Make the face of pressure foot touch with the surface of the test piece.

(1) When the scale is graduated with IRHD, apply contact force to the plunger for 5 s, and adjust the scale to be 100. Then, apply indenting force for 30 s, and read directly hardness by IRHD.

(2) When the scale is graduated with indented depth, apply contact force to the plunger for 5 s, and read the scale. Then, apply indenting force for 30 s, and read the scale. Calculate the difference between indentation by contact force and that by indenting force, and make this the indented depth *D*. Convert the value of *D* into IRHD making use of Table 3, Table 4, and Table 5.

While applying force, the slight vibration may be applied on the testing apparatus by a vibrating device to overcome the friction. Carry out measurements at 3 or 5 new points on a test piece at every measurement.

4.6 Arrangement of test results Round off the median of 3 or 5 measurements to whole number according to **JIS Z 8401**, and mark the sign IRHD after it. In case of standard hardness, after it mark "/" together with letter "S", and then mark "/" with sign as H, N, M, or L, which means testing method. In case of apparent hardness, after sign of IRHD mark "/" together with sign as H, N, M, or L, which means testing method.

Example 1 50 IRHD/S/N: means that standard test piece is measured by N method of international rubber hardness test, and standard hardness is 50 IRHD.

Example 2 50 IRHD/M: means that nonstandard test piece is measured by M method of international rubber hardness test, and apparent hardness is 50 IRHD.

4.7 Record On test result, the following items shall be recorded.

(1) Test result

(2) Shape and dimensions of test piece (whether standard test piece or nonstandard one; in case of nonstandard, whether curved surface or not; and in case of piled one, the number of piled pieces and its thickness)

(3) Sampling and preparation methods of test pieces

(4) Test temperature

(5) Other items specially needed

5 Durometer hardness test

5.1 Purpose This test shall be carried out to measure durometer hardness of vulcanized rubber.

5.2 Range of measurement The measuring range of this test is decided according to the hardness of test piece at every testing method. The measuring range of each testing method is as follows.

(1) **Type D durometer** The measuring range of type D durometer hardness is the range over A90 by type A durometer. When less than D20, measure by type A durometer.

(2) **Type A durometer** The measuring range of type A durometer hardness is from A10 to A90, and when over A90, measure by type D durometer. When less than A20, measure by type E durometer.

(3) **Type E durometer** The measuring range of type E durometer hardness is the range of less than A20 by type A durometer.

5.3 Testing apparatus

5.3.1 Outline of testing apparatus The testing apparatus is composed of the face of pressure foot by which the surface of a test piece is pressed, indentor which protrudes from a central hole of face of pressure foot by action of a spring, and the graduation which indicates the distance (indenting depth) of indentor rejected by rubber cushion and which represents hardness itself.

5.3.2 Face of pressure foot The face of pressure foot is perpendicular to the indentor, and its center has a hole for the indentor. The diameter of the hole, in case of type D and type A durometer, is $3.0^{+0.2}_{-0.5}$ mm, and in case of type E durometer, (5.4 ±0.2) mm.

On the face of pressure foot, the distance from any place of its outer edge to the center of an indentor shall be, in case of type D and type A durometer, 6 mm or more, and in case of type E durometer, 7 mm or more.

5.3.3 Indentor The material of indentor shall be abrasion resistant and corrosion resistant, and it shall be accurately fixed at center of the hole of face of pressure foot. Its shape and dimensions are indicated in Fig. 1 for type D durometer, in Fig. 2 for type A durometer, and in Fig. 3 for type E durometer.

Unit : mm

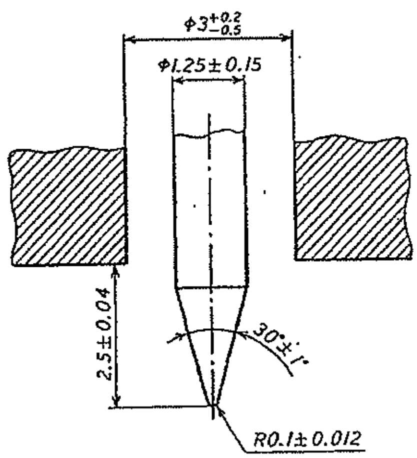


Fig. 1 Indentor for type D durometer

Unit : mm

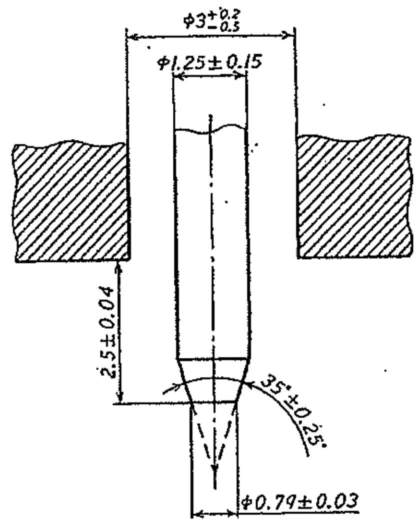


Fig. 2 Indentor for type A durometer

Unit : mm

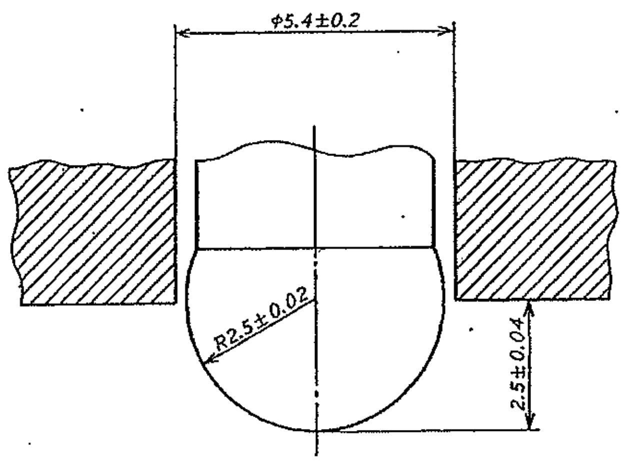


Fig. 3 Indentor for type E durometer

5.3.4 Scale When the scale indicates 0 (full protrusion), the point of the indentor shall protrude by (2.50 ±0.04) mm beyond the face of the pressure foot.

When the scale indicates 100 (nil protrusion), the face of the pressure foot is in firm contact with a flat piece of glass, i.e. the point of the indentor shall be positioned on the same plane with the face of the pressure foot. The scale shall be graduated with equal intervals in the range between 0 to 100.

5.3.5 Spring There must be the following relation between the force of spring and the scale, that is, the durometer hardness.

(1) **Type D durometer**

$$W_D = 444.5H_D\{w_D = 45.33H_D\}$$

where, W_D : force of spring of type D durometer (mN)

w_D : force of spring of type D durometer {gf}

H_D : hardness of type D durometer

(2) **Type A and type E durometer**

$$W_A = 550 + 75H_A\{w_A = 56.1 + 7.65H_A\}$$

where, W_A : force of spring of type A or type E durometer (mN)

w_A : force of spring of type A or type E durometer {gf}

H_A : hardness of type A or type E durometer

The tolerance of force shall be, in case of type D durometer, ±440 mN {±44.9 gf}, and in case of type A and type E durometer, ±80 mN {±8.16 gf}.

5.3.6 Calibration of spring Hold vertically the end point of indentor of a durometer on a balance not to give any interference between the balance and face of pressure foot, via a spacer (see Fig. 4). The cylindrical spacer with 2.5 mm height, in case of type D and type A durometer, measuring 1.25 mm in diameter, and in case of type E durometer, measuring 3 mm in diameter, has a wineglass shape where an indentor is to touch, in order to smoothly receive the end point of the indentor. Place a tare on the balance against the weight of the spacer. Place counterweight to get suitable scale, and confirm that the force (mN) shown here stays within the tolerance of specified force in **5.3.5**. Carry out the above calibration using suitable scale interval.

The calibration of spring of a durometer may be done with an electrobalance other than chemical balance shown in Fig. 4. In this case, the measuring sensitivity of the force at end point of an indentor shall be, in case of type D durometer, 44 mN {4.5 gf} or less, and in case of type A and type E durometer, 8 mN {0.82 gf} or less.

The following method is permissible; place upside down the durometer, and directly apply the load on its indentor by counterweight. Provided that the correction about the mass of parts inside of the durometer shall be considered to prevent the discrepancy between this method and the method by Fig. 4. In this case, the accuracy on the mass of counterweight shall be ±4.5 g or less in case of type D durometer and ±0.82 g or less in case of type A and type E durometer.

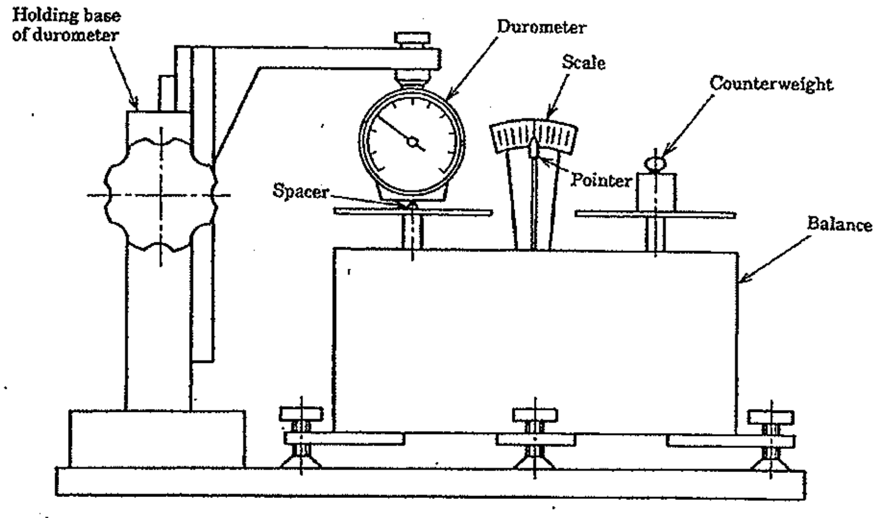


Fig. 4 Example of calibration apparatus of spring

5.4 Test piece

5.4.1 Shape and dimensions of test pieces The thickness of a test piece for type D and type A durometer is 6 mm or more. When it is less than 6 mm, pile them to make 6 mm or more for measurement. The thickness of a test piece for type E durometer is 10 mm or more, and in case of less than 10 mm, pile them to make 10 mm or more. The number of test pieces to pile shall be at most 3, and each of them shall have 2 mm or more thickness. The test result brought by piled up test piece doesn't generally coincide with the result by solid test piece(13). The lateral size of test piece shall be large enough to measure at the point where the end point of an indentor is apart 12 mm or more from the edge of the test piece.

Furthermore, the test piece shall have smooth surface spacious enough to make close contact with face of pressure foot of a durometer(14).

Notes (13) To make comparison, it is necessary to use the test piece which has the same number for piling and the same thickness.

(14) The surface such as unsmoothed, curved, or rough, does not give satisfactory results. For specially formed surface, however, such as rubber roll, this method can be applied. In this case, the applicable limit of the durometer shall be definitely confirmed.

5.4.2 Sampling and preparation of test pieces The sampling and preparation of test pieces shall follow **6.5** of **JIS K 6250.**

5.4.3 Selection of test pieces The test pieces which contain alien matters, bubbles, or flaws shall not be used for test.

5.5 Testing method

5.5.1 Testing conditions Testing conditions shall be as follows.

(1) The standard conditions of a laboratory shall follow **6.1** of **JIS K 6250.**

(2) Storing of sample and test pieces shall follow **6.2** of **JIS K 6250**.

(3) The standard conditions of test pieces shall follow **6.3** of **JIS K 6250**.

5.5.2 Procedures Place a test piece on a rigid, hard, and flat surface. Set a durometer so as to make an indentor rectangular to the target surface of a test piece. Contact closely as swiftly as possible the face of pressure foot with the target surface of the test piece without giving a impact, and read the scale within 1 s, to find the hardness of the test piece(15). But the agreement between the parties concerned with delivery may permit to read when a definite time passed after close contacting between them. The end point of the indentor of a durometer must be apart 12 mm or more from the edge of the test piece. Unless otherwise specified, the duration from close contacting to the finish of reading shall be recorded. The measuring points shall be 5, which are apart at least 6 mm each other, and carry out measurements 5 times on these points. When hardness shown by type A durometer is over A90, employ a type D durometer. When the hardness shown by type D durometer is less than D20, employ a type A durometer. If the hardness by type A durometer is less than A10, result is inaccurate, so don't record it.

When the hardness by a type A durometer is less A20, measure it with a type E durometer.

Note (15) In order to get a good repeatability, the holding base for durometer may be used by which the durometer is vertically kept and target surface and indentor get right angle each other before measurement. In this case, it is recommended that the mass imposed on the pressing surface is 5.0 kg for type D durometer, and 1.0 kg for both type A and type E durometer.

5.6 Arrangement of test results Round off the median of 5 measurements to whole number according to **JIS Z 8401**, and mark sign D in case of type D durometer, sign A in case of type A durometer, and sign E in case of type E durometer, just before the rounded value. When the value was read when definite time passed after close contacting, mark sign "/" and then record the duration (s). When it is standard hardness, the above is followed by "/" and then by sign S.

Example 1 D85/15/S: means that standard test piece is measured by type D durometer hardness test, and the reading on standard hardness is 85 when 15 s passed after close contacting of face of pressure foot.

Example 2 A45/S: means that standard test piece is measured by type A durometer hardness test, and the reading on standard hardness is 45 within 1 s after close contacting of face of pressure foot.

Example 3 A45/15: means that nonstandard test piece is measured by type A durometer hardness test, and the reading on apparent hardness is 45 when 15 s passed after close contacting of face of pressure foot.

Example 4 E60: means that nonstandard test piece is measured by type E durometer hardness test, and the reading on apparent hardness is 60 within 1 s after close contacting of face of pressure foot.

5.7 Record On test result, the following items shall be recorded.

(1) Test result

(2) Shape and dimensions of test piece (whether standard test piece or nonstandard test piece; in case of piled up test piece, the number of piled pieces, and its thickness)

(3) Sampling and preparation methods of test pieces

(4) Other items specially needed

6 IRHD pocket hardness test

6.1 Purpose This test shall be carried out to measure the international rubber hardness degree of vulcanized rubber by IRHD pocket hardness meter, and abbreviated P method.

6.2 Testing apparatus

6.2.1 Outline of testing apparatus The testing apparatus is composed of a face of pressure foot to press the surface of a test piece, indentor which protrudes from a central hole of face of pressure foot by action of a spring, and a mechanism indicating the protruded length of the indentor.

6.2.2 Face of pressure foot The face of pressure foot, measuring (20 ±2.5) mm sided square, has a hole with 2.0 mm to 3.0 mm diameter at its center.

6.2.3 Indentor The end of the indentor shall make a hemisphere with 1.55 mm to 1.60 mm diameter.

6.2.4 Indicating mechanism The indicating mechanism shows the protruded length of an indentor from face of pressure foot, and it shall have been calibrated to read directly the international rubber hardness degree by IRHD. When the longest protruded length of 1.65 mm is given, it must show 28 IRHD, and when the face of pressure foot is let contact with a flat glass, that is, no protruded, it must show 100 IRHD.

6.2.5 Spring Spring can apply constant force of (2.65 ±0.15) N {(270.3 ±15.3) gf} to an indentor in the range from 28 IRHD to 100 IRHD.

6.2.6 Calibration of hardness meter IRHD pocket hardness meter shall be calibrated and adjusted using a standard rubber block whose international rubber hardness degree has been known. Only when the standard rubber block cannot be used, it is preferably calibrated with mechanical method.

Press the IRHD pocket hardness meter on a flat glass plate, and adjust the scale to get 100 IRHD. Making use of a set of standard rubber blocks from 30 IRHD to 90 IRHD, calibrate IRHD pocket hardness meter. The set of standard rubber blocks is stored in a container with a suitable cover after being sprinkled with talc powder, in order to prevent the influences by light, heat, oil, or grease. It consists of at least 6 test pieces. These standard blocks must be calibrated with the international rubber hardness test specified in 4 at intervals not exceeding six months. It is advisable that the IRHD pocket hardness meter, which is used daily, is calibrated at least once a week with standard rubber block.

Remarks : When IRHD pocket hardness meter is calibrated with mechanical method or adjusted, the instruction manual issued by the manufacturer shall be depended.

6.3 Test piece

6.3.1 Shape and dimensions of test pieces The thickness of a test piece shall be 6 mm or more. When it is less than 6 mm, the test piece which was prepared by piling up to 6 mm or more can be used, but the number of piling up shall be 3 or less, and each of them shall have 2 mm or more thickness. The test result comes from piled test piece does not usually coincide with the test result by solid test piece(13). The lateral dimension of a test piece shall be large enough to measure at the point where the end point of an indentor is apart 12 mm or more from the edge of the test piece.

Test pieces shall have flat surface which is spacious to closely contact with the face of pressure foot of a hardness meter(16).

Note (16) The surface such as unsmoothed, curved, or rough, does not give satisfactory results. For specially formed surface, however, such as rubber roll, this method can be applied. In this case, the applicable limit of the IRHD pocket hardness meter shall be definitely confirmed.

6.3.2 Sampling and preparation of test pieces The sampling and preparation of test pieces shall follow **6.5** of **JIS K 6250**.

6.3.3 Selection of test pieces The test pieces which contain alien matters, bubbles, or flaws shall not be used for test.

6.4 Testing method

6.4.1 Testing conditions Testing conditions shall be as follows.

(1) The standard conditions of a laboratory shall follow **6.1** of **JIS K 6250**.

(2) Storing of sample and test pieces shall follow **6.2** of **JIS K 6250**.

(3) The standard conditions of test pieces shall follow **6.3** of **JIS K 6250**.

6.4.2 Procedures Place a test piece on a rigid, hard, and flat surface. Set an IRHD pocket hardness meter so as to make an indentor rectangular to the target surface of a test piece. Contact closely as swiftly as possible the face of pressure foot with the target surface of the test piece without giving a impact, and read the scale within 1 s, to find the hardness of the test piece. The end point of the indentor of an IRHD pocket hardness meter must be apart 12 mm or more from the edge of the test piece. Unless otherwise specified, read the value within 1 s after close contacting, but if the reading after special duration is specified, follow that specification. In this case, the duration from close contacting to the finish of reading shall be recorded. The measuring points shall be 5, which are apart at least 6 mm each other, and carry out measurements 5 times on these points.

6.5 Arrangement of test results Round off the median of 5 measurements to whole number according to **JIS Z 8401**, then mark sign IRHD after the value, and in case of standard hardness, after the value mark sign "/", then sign S, then again sign "/" and last sign P which means testing method. In case of apparent hardness, mark sign "/" after sign IRHD, then mark sign P which means testing method.

Example 1 50 IRHD/S/P: means that standard test piece is measured by IRHD pocket hardness meter, and the standard hardness is 50 IRHD.

Example 2 50 IRHD/P: means that nonstandard test piece is measured by IRHD pocket hardness meter, and the apparent hardness is 50 IRHD.

6.6 Record On test result, the following items shall be recorded.

(1) Test result

(2) Shape and dimensions of test piece (whether standard test piece or nonstandard test piece; in case of piled up test piece, the number of piled pieces, and its thickness)

(3) Sampling and preparation methods of test pieces

(4) Other items specially needed

Related standards :

ISO 7267/1 : 1986 *Rubber-covered rollers—Determination of apparent hardness—Part 1 : IRHD method*

ISO 7267/2 : 1986 *Rubber-covered rollers—Determination of apparent hardness—Part 2 : Shore-type durometer method*

Informative reference

International rubber hardness testing method for curved test piece

Introduction This Informative reference states the international rubber hardness testing method for curved test piece, and does not make a part of Standard.

1 Purpose This test shall be carried out to measure international rubber hardness degree of a test piece of vulcanized rubber whose target surface makes a curved surface. The measured values obtained by this method are always treated as an apparent hardness.

Remarks : The standards cited in this Informative reference are listed as follows.

ISO 48 : 1994 *Rubber, vulcanized or thermoplastic—Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 7267/1 : 1986 *Rubber-covered rollers—Determination of apparent hardness—Part 1 : IRHD method*

ISO 7267/2 : 1986 *Rubber-covered rollers—Determination of apparent hardness—Part 2 : Shore-type durometer method*

2 Type of testing method

(1) CH method (normal size curved surface test for high hardness)

(2) CN method (normal size curved surface test for normal hardness)

(3) CM method (microsize curved surface test for normal hardness)

(4) CL method (normal size curved surface test for low hardness)

3 Scope CH method, CN method, CM method, and CL method are the modified H method, N method, M method, and L method for the purpose of making them applicable to the test piece whose target surface is curved, and there are the following two cases(1).

(1) Test piece or sample is large enough to place the hardness testing apparatus on it.

(2) Test piece or sample is so small that it must be placed on a holding base together with a hardness testing apparatus. The case where the sample is put on a flat sample base which makes one body with a testing apparatus, is included in this case.

Note (1) Generally, these tests are carried out directly on products, so that the thickness of rubber is not constant, and in many cases, the lateral distance from the end ball of a plunger to the edge of sample is smaller than the smallest distance shown in **4.4.3** in the body of this Standard, and the influence owing to the distance from the edge is not negligible.

Therefore, the measured values resulted from these methods don't coincide with the values obtained by the measurements of the plate-type test pieces with flat parallel surfaces and the same thickness as that of standard test pieces or products which are specified in H method, N method, M method and L method.

This means that, the results obtained by measuring curved surface are the peculiar measurements which are applicable only to the test pieces or the products having special shape and special dimensions and further being kept in special method. In extreme case, these measured values show discrepancy of 10 IRHD from the standard hardness. The measured values on the surface buffed to eliminate covered cloth or treated specially, shows a little difference value from the value on flat surface which has been finished with molding.

4 Testing apparatus

4.1 General matters Basically, testing apparatus follows **4.3** of the body of this Standard, but the following gives difference.

4.2 Testing apparatus for cylindrical surface of 50 mm or more radius As shown in Informative reference Fig. 1, the bottom base of the testing apparatus has a hole through which annular pressure foot can penetrate, for the measurement even when sample is put under the base.

There are two cylindrical surfaces which are parallel each other under the base, and these are parallel to the horizontal surface of the base. The diameter of these cylinders and the distance between them shall be suitable for setting up testing apparatus on the target curved surface of sample. Alternatively, the base, on which adjustable legs with universal joints are attached to comply with the target curved surface, may be used.

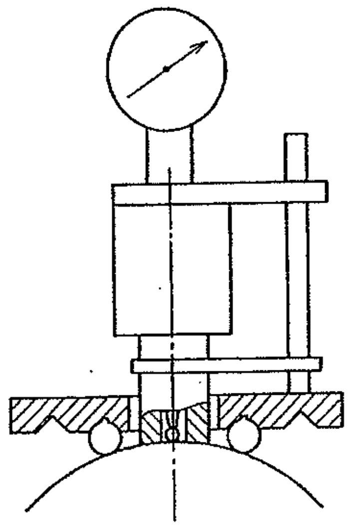
4.3 Testing apparatus for two-way curved surface of 50 mm or more radius The testing apparatus with adjustable legs with universal joints shown in **4.2** can be used.

4.4 Testing apparatus for cylindrical surface and two-way curved surface of 4 mm to 50 mm radius When target surface is too small to set a testing apparatus on it, as shown in Informative reference Fig. 2, fix test piece or sample using a special jig, V-block, or the like, and set the plunger to be perpendicular onto the target surface. When a small test piece is fixed on a sample table, wax may be used(2)(3).

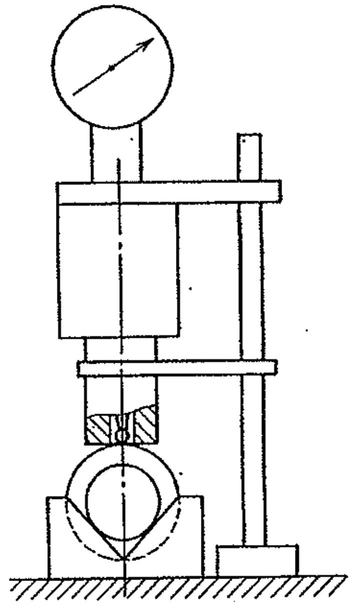
Notes (2) The testing apparatus for M method shall be generally used only for the test piece whose thickness is 4 mm or less.

(3) The testing apparatus for M method, whose sample table is forced up owing to the action of a spring, is not suitable for the large-sized test piece or sample having curved surface with large radius.

4.5 Testing apparatus for small type O-ring and curved sample of 4 mm or less radius In these cases, hold a test piece on the table of testing apparatus using a suitable jig, block, wax, or the like. Carry out measurement using a testing apparatus of M method. The test piece having the minimum radius of 0.8 mm or less cannot be measured.



Informative reference Fig. 1 Example of setting a testing apparatus for sample with large diameter



Informative reference Fig. 2 Example of setting a testing apparatus for sample with small diameter

5 Test pieces

5.1 General matters The test pieces for CH method, CN method, CM method, and CL method are the products or the pieces prepared by cutting the products. The bottom side of the test piece which has been cut out shall be held with suitable method. In case of the target surface is covered with cloth, it must be buffed before testing. In order to recover it from the influence by buffing, allow it to stand for 16 h or more under standard condition of laboratory, and then carry out conditioning under standard condition according to (3) of **4.5.1** in the body of this Standard. This duration may be included in the duration for recovering.

5.2 Sampling and preparation of test pieces The sampling and preparation of test pieces shall follow **4.4.4** in the body of this Standard.

5.3 Selection of test pieces The selection of test pieces shall follow **4.4.5** in the body of this Standard.

6 Testing method The testing method shall follow **4.5** in the body of this Standard.

7 Arrangement of test results Round off the median of 3 or 5 measurements to whole number according to **JIS Z 8401**, and then mark sign IRHD after the value. After that, mark sign "/", and then mark CH, CN, CM, or CL which means testing method.

Example : 50 IRHD/CM: means that a curved test piece is measured by CM method of international rubber hardness curved-surface test, and the hardness is 50 IRHD.

8 Record On test result, the following items shall be recorded.

(1) Test result

(2) Shape and dimensions of test pieces

(3) Sampling and preparation methods of test piece

(4) Test temperature

(5) Other items specially needed

Errata for JIS (English edition) are printed in *Standardization Journal*, published monthly by the Japanese Standards Association, and also provided to subscribers of JIS (English edition) in *Monthly Information*.

Errata will be provided upon request, please contact:
Standardization Promotion Department, Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN
TEL. 03-3583-8002 FAX. 03-3583-0462

TAB 42

Designation: D 2240 – 05

INTERNATIONAL

Standard Test Method for Rubber Property—Durometer Hardness[1]

This standard is issued under the fixed designation D 2240; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers twelve types of rubber hardness measurement devices known as durometers: Types A, B, C, D, DO, E, M, O, OO, OOO, OOO-S, and R. The procedure for determining indentation hardness of substances classified as thermoplastic elastomers, vulcanized (thermoset) rubber, elastomeric materials, cellular materials, gel-like materials, and some plastics is also described.

1.2 This test method is not equivalent to other indentation hardness methods and instrument types, specifically those described in Test Method D 1415.

1.3 This test method is not applicable to the testing of coated fabrics.

1.4 All materials, instruments, or equipment used for the determination of mass, force, or dimension shall have traceability to the National Institute for Standards and Technology, or other internationally recognized organizations parallel in nature.

1.5 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only. Many of the stated dimensions in SI are direct conversions from the U. S. Customary System to accommodate the instrumentation, practices, and procedures that existed prior to the Metric Conversion Act of 1975.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 374 Test Methods for Thickness of Solid Electrical Insulation

D 618 Practice for Conditioning Plastics for Testing

D 785 Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials

D 1349 Practice for Rubber—Standard Temperatures for Testing

D 1415 Test Method for Rubber Property—International Hardness

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries

F 1957 Test Method for Composite Foam Hardness-Durometer Hardness

2.2 *ISO Standard:*[3]

ISO/IEC 17025: 1999 General Requirements for the Competence of Testing and Calibration Laboratories

3. Summary of Test Method

3.1 This test method permits hardness measurements based on either initial indentation or indentation after a specified period of time, or both. Durometers with maximum reading indicators used to determine maximum hardness values of a material may yield lower hardness when the maximum indicator is used.

3.2 The procedures for Type M, or micro hardness durometers, accommodate specimens that are, by their dimensions or configuration, ordinarily unable to have their durometer hardness determined by the other durometer types described. Type M durometers are intended for the testing of specimens having a thickness or cross-sectional diameter of 1.25 mm (0.050 in.) or greater, although specimens of lesser dimensions may be successfully accommodated under the conditions specified in Section 6, and have a Type M durometer hardness range between 20 and 90. Those specimens which have a durometer hardness range other than specified shall use another suitable procedure for determining durometer hardness.

4. Significance and Use

4.1 This test method is based on the penetration of a specific type of indentor when forced into the material under specified conditions. The indentation hardness is inversely related to the penetration and is dependent on the elastic modulus and viscoelastic behavior of the material. *The geometry of the*

---

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.10 on Physical Testing.

Current edition approved Aug. 15, 2005. Published September 2005. Originally approved in 1964. Last previous edition approved in 2004 as D 2240–04$^{\epsilon 1}$.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from International Organization for Standardization (ISO), 1 rue de Varembé, Case postale 56, CH-1211, Geneva 20, Switzerland.

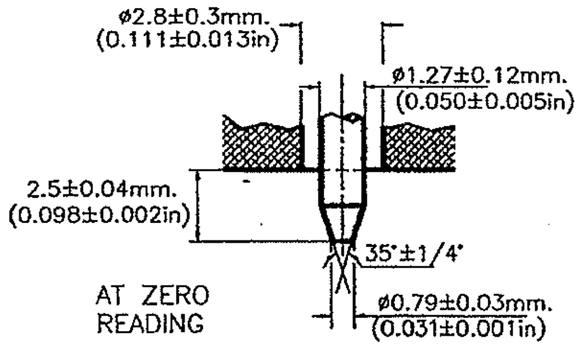


FIG. 1 (a) Type A and C Indentor

indentor and the applied force influence the measurements such that no simple relationship exists between the measurements obtained with one type of durometer and those obtained with another type of durometer or other instruments used for measuring hardness. This test method is an empirical test intended primarily for control purposes. No simple relationship exists between indentation hardness determined by this test method and any fundamental property of the material tested. For specification purposes, it is recommended that Test Method D 785 be used for materials other than those described in 1.1.

5. Apparatus

5.1 *Hardness Measuring Apparatus, or Durometer, and an Operating Stand,* Type 1, Type 2, or Type 3 (see 5.1.2) consisting of the following components:

5.1.1 *Durometer:*

5.1.1.1 *Presser Foot,* the configuration and the total area of a durometer presser foot may produce varying results when there are significant differences between them. It is recommended that when comparing durometer hardness determinations of the same type (see 4.1), that the comparisons be between durometers of similar presser foot configurations and total area, and that the presser foot configuration and size be noted in the Hardness Measurement Report (see 10.2.4 and 5.1.1.3).

5.1.1.2 *Presser Foot,* Types A, B, C, D, DO, E, O, OO, OOO, and OOO-S, with an orifice (to allow for the protrusion of the indentor) having a diameter as specified in Fig. 1 (a, b, c, d, e, f, and g), with the center a minimum of 6.0 mm (0.24 in.) from any edge of the foot. When the presser foot is not of a flat circular design, the area shall not be less than 500 mm^2 (19.7 in.^{2}).

NOTE 1—The Type OOO and the Type OOO-S, designated herein, differ in their indentor configuration, spring force, and the results obtained. See Table 1 and Fig. 1 (e and g).

5.1.1.3 *Presser Foot*—flat circular designs designated as Type *xR*, where *x* is the standard durometer designation and *R* indicates the flat circular press foot described herein, for example, Type *aR*, *dR*, and the like. The presser foot, having a centrally located orifice (to allow for the protrusion of the indentor) of a diameter as specified in Fig. 1 (a through g). The flat circular presser foot shall be 18 ± 0.5 mm (0.71 ± 0.02 in.) in diameter. These durometer types shall be used in an operating stand (see 5.1.2).

(a) Durometers having a presser foot configuration other than that indicated in 5.1.1.3 shall not use the Type *xR* designation, and it is recommended that their presser foot configuration and size be stated in the Hardness Measurement Report (see 10.2.4).

5.1.1.4 *Presser Foot, Type M,* with a centrally located orifice (to allow for the protrusion of the indentor), having a diameter as specified in Fig. 1 (d), with the center a minimum of 1.60 mm (0.063 in.) from any edge of the flat circular presser foot. The Type M durometer shall be used in a Type 3 operating stand (see 5.1.2.4).

5.1.1.5 *Indentor,* formed from steel rod and hardened to 500 HV10 and shaped in accordance with Fig. 1 (a, b, c, d, e, or g), polished over the contact area so that no flaws are visible under 20× magnification, with an indentor extension of 2.50 ± 0.04 mm (0.098 ± 0.002 in.).

5.1.1.6 *Indentor, Type OOO-S,* formed from steel rod and hardened to 500 HV10, shaped in accordance with Fig. 1 (f), polished over the contact area so that no flaws are visible under 20× magnification, with an indentor extension of 5.00 ± 0.04 mm (0.198 ± 0.002 in.).

5.1.1.7 *Indentor, Type M,* formed from steel rod and hardened to 500 HV10 and shaped in accordance with Fig. 1 (d), polished over the contact area so that no flaws are visible under 50× magnification, with an indentor extension of 1.25 ± 0.02 mm (0.049 ± 0.001 in.).

5.1.1.8 *Indentor Extension Indicator,* analog or digital electronic, having a display that is an inverse function of the indentor extension so that:

(1) The display shall indicate from 0 to 100 with no less than 100 equal divisions throughout the range at a rate of one hardness point for each 0.025 mm (0.001 in.) of indentor movement.

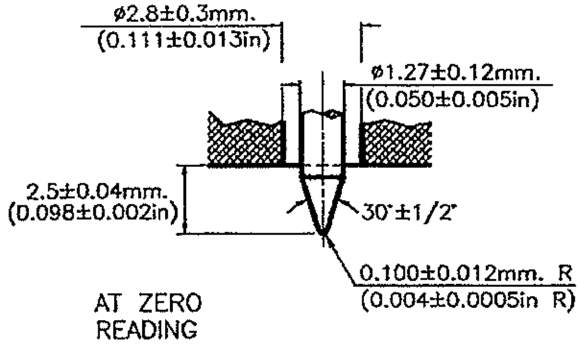


FIG. 1 (b) Type B and D Indentor *(continued)*

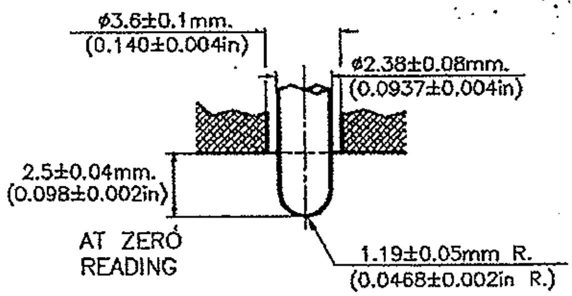


FIG. 1 (c) Type O, DO, and OO Indentor *(continued)*

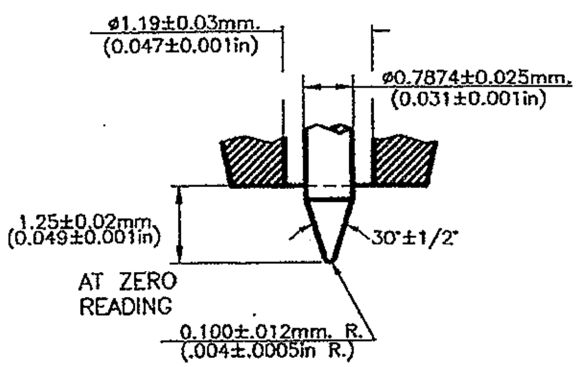


FIG. 1 (d) Type M Indentor *(continued)*

(2) The display for Type OOO-S durometers shall indicate from 0 to 100 with no less than 100 equal divisions throughout the range at a rate of one hardness point for each 0.050 mm (0.002 in.) of indentor movement,

(3) The display for Type M durometers shall indicate from 0 to 100 with no less than 100 equal divisions at a rate of one hardness point for each 0.0125 mm (0.0005 in.) of indentor movement, and

(4) In the case of analog dial indicators having a display of 360°, the points indicating 0 and 100 may be at the same point on the dial and indicate 0, 100, or both.

5.1.1.9 *Timing Device (optional)*, capable of being set to a desired elapsed time, signaling the operator or holding the hardness reading when the desired elapsed time has been reached. The timer shall be automatically activated when the presser foot is in contact with the specimen being tested, for example, the initial indentor travel has ceased. Digital electronic durometers may be equipped with electronic timing devices that shall not affect the indicated reading or determinations attained by more than one-half of the calibration tolerance stated in Table 1.

5.1.1.10 *Maximum Indicators (optional)*, maximum indicating pointers are auxiliary analog indicating hands designed to remain at the maximum hardness value attained until reset by

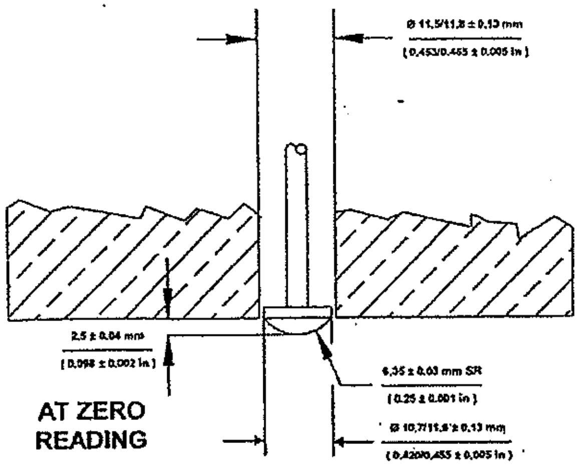


FIG. 1 (e) Type OOO Indentor *(continued)*

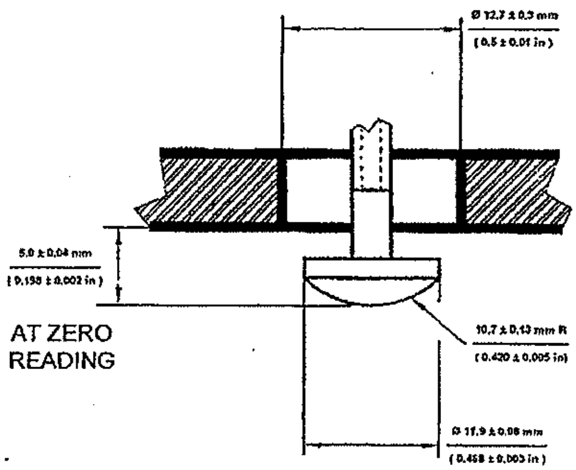


FIG. 1 (f) Type OOO-S Indentor *(continued)*

the operator. Electronic maximum indicators are digital displays electronically indicating and maintaining the maximum value hardness valued achieved until reset by the operator.

5.1.1.11 Analog maximum indicating pointers have been shown to have a nominal effect on the values attained, however, this effect is greater on durometers of lesser total mainspring loads; for example, the effect of a maximum indicating pointer on Type D durometer determinations will be less than those determinations achieved using a Type A durometer. Analog style durometers may be equipped with maximum indicating pointers. The effect of a maximum indicating pointer shall be noted at the time of calibration in the calibration report (see 10.1.5), and when reporting hardness determinations (see 10.2.4). Analog Type M, OO, OOO, and Type OOO-S durometers shall not be equipped with maximum indicating pointers.

5.1.1.12 Digital electronic durometers may be equipped with electronic maximum indicators that shall not affect the indicated reading or determinations attained by more than one half of the spring calibration tolerance stated in Table 1.

5.1.1.13 *Calibrated Spring*, for applying force to the indentor, in accordance with Fig. 1 (a through g) and capable of applying the forces as specified in Table 1.

5.1.2 *Operating Stand* (Fig. 2):

5.1.2.1 Type 1, Type 2, and Type 3 shall be capable of supporting the durometer presser foot surface parallel to the specimen support table (Fig. 3) throughout the travel of each. The durometer presser foot to specimen support table parallelism shall be verified each time the test specimen support table is adjusted to accommodate specimens of varying dimensions. This may be accomplished by applying the durometer presser foot to the point of contact with the specimen support table and making adjustments by way of the durometer mounting assembly or as specified by the manufacturer.

5.1.2.2 *Operating Stand, Type 1* (specimen to indentor type), shall be capable of applying the specimen to the indentor in a manner that minimizes shock.

5.1.2.3 *Operating Stand, Type 2* (indentor to specimen type), shall be capable of controlling the rate of descent of the indentor to the specimen at a maximum of 3.20 mm/s (0.125

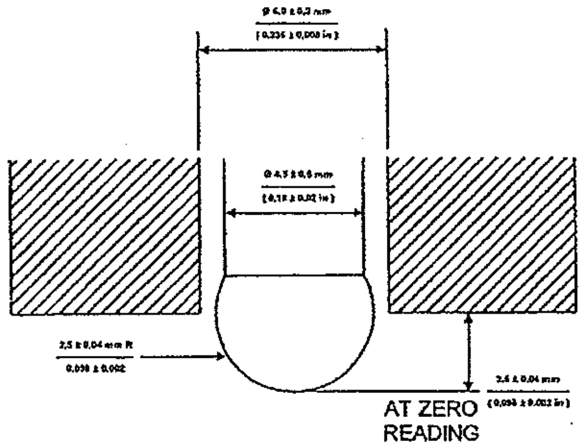


FIG. 1 (g) Type E Indentor *(continued)*

TABLE 1 Durometer Spring Force Calibration[A]
All Values are in N

Indicated Value	Type A, B, E, O	Type C, D, DO	Type M	Type OO, OOO	Type OOO-S
0	0.55	0	0.324	0.203	0.167
10	1.3	4.445	0.368	0.294	0.343
20	2.05	8.89	0.412	0.385	0.520
30	2.8	13.335	0.456	0.476	0.696
40	3.55	17.78	0.5	0.566	0.873
50	4.3	22.225	0.544	0.657	1.049
60	5.05	26.67	0.589	0.748	1.226
70	5.8	31.115	0.633	0.839	1.402
80	6.55	35.56	0.677	0.93	1.579
90	7.3	40.005	0.721	1.02	1.755
100	8.05	44.45	0.765	1.111	1.932
N/durometer unit	0.075	0.4445	0.0044	0.00908	0.01765
Spring Calibration Tolerance	± 0.075 N	± 0.4445 N	± 0.0176 N	± 0.0182 N	± 0.0353 N

[A] Refer to 5.1.1.3 for the Type *xR* designation.

in./s) and applying a force sufficient to overcome the calibrated spring force as shown in Table 1.

5.1.2.4 *Operating Stand, Type 3* (indentor to specimen type), hydraulic dampening, pneumatic dampening, or electro-mechanical (required for the operation of Type M durometers) shall be capable of controlling the rate of descent of the indentor to the specimen at a maximum of 3.2 mm/s (0.125 in./s) and applying a force sufficient to overcome the calibrated spring force as shown in Table 1. Manual application, Type 1 or Type 2 operating stands are not acceptable for Type M durometer operation.

5.1.2.5 The entire instrument should be plumb and level, and resting on a surface that will minimize vibration. Operating the instrument under adverse conditions will negatively affect the determinations attained.

5.1.2.6 *Specimen Support Table*, (Fig. 3) integral to the operating stand, and having a solid flat surface. The specimen support platform may have orifices designed to accept various inserts or support fixtures (Fig. 3) to provide for the support of irregularly configured specimens. When inserts are used to support test specimens, care must be taken to align the indentor to the center of the insert, or the point at which the indentor is to contact the specimen. Care should be exercised to assure that the indentor does not abruptly contact the specimen support table as damage to the indentor may result.

6. Test Specimen

6.1 The test specimen, herein referred to as "specimen" or "test specimen" interchangeably, shall be at least 6.0 mm (0.24 in.) in thickness unless it is known that results equivalent to the 6.0-mm (0.24-in.) values are obtained with a thinner specimen.

6.1.1 A specimen may be composed of plied pieces to obtain the necessary thickness, but determinations made on such specimens may not agree with those made on solid specimens, as the surfaces of the plied specimens may not be in complete contact. The lateral dimensions of the specimen shall be sufficient to permit measurements at least 12.0 mm (0.48 in.) from any edge, unless it is known that identical results are obtained when measurements are made at a lesser distance from an edge.

6.1.2 The surfaces of the specimen shall be flat and parallel over an area to permit the presser foot to contact the specimen over an area having a radius of at least 6.0 mm (0.24 in.) from the indentor point. The specimen shall be suitably supported to provide for positioning and stability. *A suitable hardness*

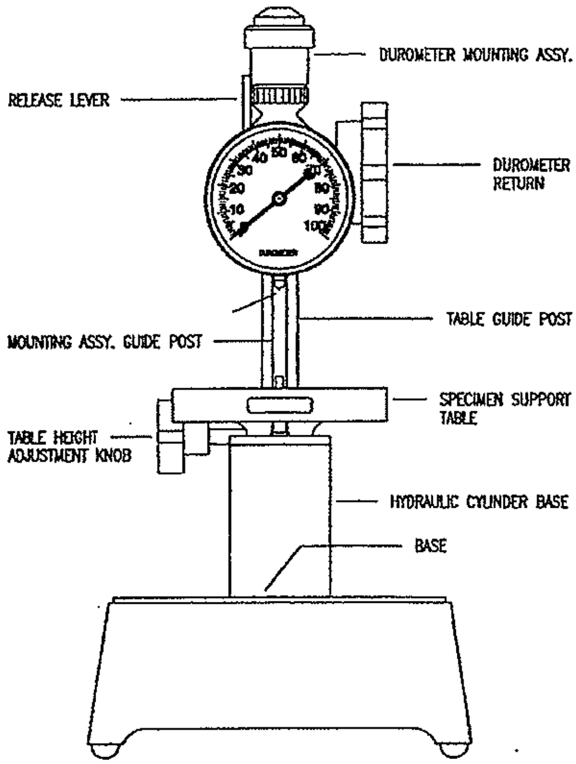


FIG. 2 Durometer Operating Stand

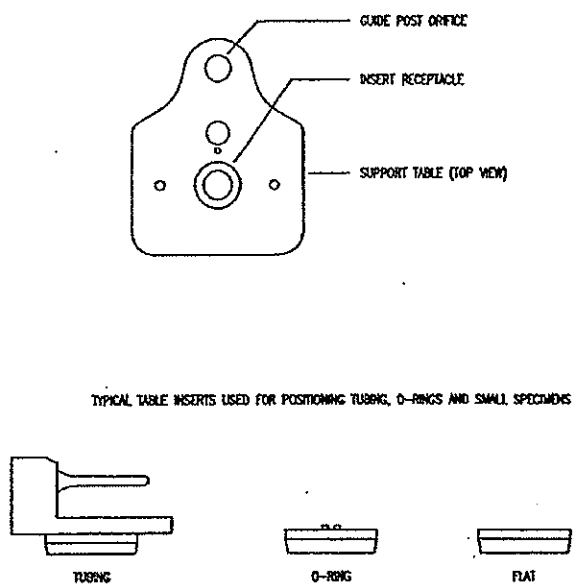


FIG. 3 Small Specimen Support Table

determination cannot be made on an uneven or rough point of contact with the indentor.

6.2 Type OOO, OOO-S, and M test specimens should be at least 1.25 mm (0.05 in.) in thickness, unless it is known that results equivalent to the 1.25-mm (0.05-in.) values are obtained with a thinner specimen.

6.2.1 A Type M specimen that is not of a configuration described in 6.2.2 may be composed of plied pieces to obtain

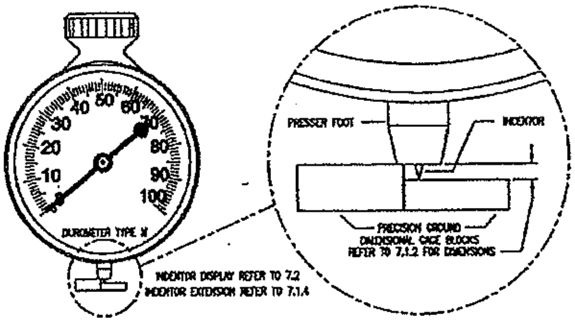


FIG. 4 Detail of Indentor Extension and Display Adjustment

the necessary thickness, but determinations made on such specimens may not agree with those made on solid specimens because the surfaces of the plied specimens may not be in complete contact. The lateral dimensions of the specimen should be sufficient to permit measurements at least 2.50 mm (0.10 in.) from any edge unless it is known that identical results are obtained when measurements are made at lesser distance from an edge. *A suitable hardness determination cannot be made on an uneven or rough point of contact with the indentor.*

6.2.2 The Type M specimen, when configured as an o-ring, circular band, or other irregular shape shall be at least 1.25 mm (0.05 in.) in cross-sectional diameter, unless it is known that results equivalent to the 1.25-mm (0.05-in.) values are obtained with a thinner specimen. The specimen shall be suitably supported in a fixture (Fig. 3) to provide for positioning and stability.

6.3 The minimum requirement for the thickness of the specimen is dependent on the extent of penetration of the indentor into the specimen; for example, thinner specimens may be used for materials having higher hardness values. The minimum distance from the edge at which measurements may be made likewise decreases as the hardness increases.

7. Calibration

7.1 *Indentor Extension Adjustment Procedure*:

7.1.1 Place precision ground dimensional blocks (Grade B or better) on the support table and beneath the durometer presser foot and indentor. Arrange the blocks so that the durometer presser foot contacts the larger block(s) and the indentor tip just contacts the smaller block (Fig. 4). It is necessary to observe the arrangement of the blocks and the presser foot/indentor under a minimum of 20× magnification to assure proper alignment.

7.1.2 Indentor extension and shape shall be in accordance with 5.1.1.5, 5.1.1.6, or 5.1.1.7, respective to durometer type. See Fig. 1 (a through g). Examination of the indentor under 20× magnification, 50× for Type M indentors, is required to examine the indentor condition. Misshapen or damaged indentors shall be replaced.

7.1.3 A combination of dimensional gage blocks shall be used to achieve a difference of 2.54 + 0.00/–0.0254 mm (0.100 + 0.00/–0.001 in.) between them. For Type OOO-S durometers, the gage block dimensions are 5.08 + 0.00/–0.0508 mm (0.200 + 0.00/–0.002 in.). For Type M durometers, the gage block dimensions are 1.27 + 0.0/–0.0127 mm (0.050 + 0.00/–0.0005 in.) between them (Fig. 4).

7.1.4 Carefully lower the durometer presser foot until it contacts the largest dimensional block(s), the indentor tip should just contact the smaller block, verifying full indentor extension.

7.1.5 Adjust the indentor extension to 2.50 ± 0.04 mm (0.098 ± 0.002 in.). For Type OOO-S durometers, adjust the indentor extension to 5.0 ± 0.04 mm (0.198 ± 0.002 in.). For Type M durometers, adjust the indentor extension to 1.25 ± 0.02 mm (0.049 ± 0.001 in.), following the manufacturer's recommended procedure.

7.1.5.1 When performing the procedures in 7.1, care should be used so as not to cause damage to the indentor tip. Fig. 4 depicts a suitable arrangement for gaging indentor extension.

7.1.6 Parallelism of the durometer presser foot to the support surface, and hence the dimensional gage blocks, at the time of instrument calibration, may be in accordance with Test Methods D 374, Machinist's Micrometers, or otherwise accomplished in accordance with the procedures specified by the manufacturer.

7.2 *Indentor Display Adjustment*:

7.2.1 After adjusting the indentor extension as indicated in 7.1, use a similar arrangement of dimensional gage blocks to verify the linear relationship between indentor travel and indicated display at two points: 0 and 100. Following the manufacturer's recommendations, make adjustments so that:

7.2.2 The indicator displays a value equal to the indentor travel measured to within:

–0.0 +1.0 durometer units measured at 0;

±0.50 durometer units measured at 100;

±1 durometer units at all other points delineated in 7.4.

7.2.3 Each durometer point indicated is equal to 0.025 mm (0.001 in.) of indentor travel, except for:

7.2.3.1 Type M Durometers, each indicated point is equal to 0.0125 mm (0.0005 in.) of indentor travel;

7.2.3.2 Type OOO-S Durometers, each indicated point is equal to 0.050 mm (0.002 in.) of indentor travel.

7.2.4 The indicator shall not display a value greater than 100 or less than 0 at the time of calibration.

7.2.5 Other means of determining indentor extension or indentor travel, such as optical or laser measurement methods, are acceptable. The instrumentation used shall have traceability as described in 1.4.

7.2.6 The durometer shall be supported in a suitable fashion when performing the procedures described in 7.1 and 7.2.

7.3 *Calibration Device*:

7.3.1 The durometer spring shall be calibrated by supporting the durometer in a calibrating device, see Fig. 5, in a vertical position and applying a measurable force to the indentor tip. The force may be measured by means of a balance as depicted in Fig. 5, or an electronic force cell. The calibrating device shall be capable of measuring applied force to within 0.5 % of the maximum spring force necessary to achieve 100 durometer units.

7.3.2 Care should be taken to ensure that the force is applied vertically to the indentor tip, as lateral force will cause errors in calibration. See 7.1.5.1 and 7.1.6.

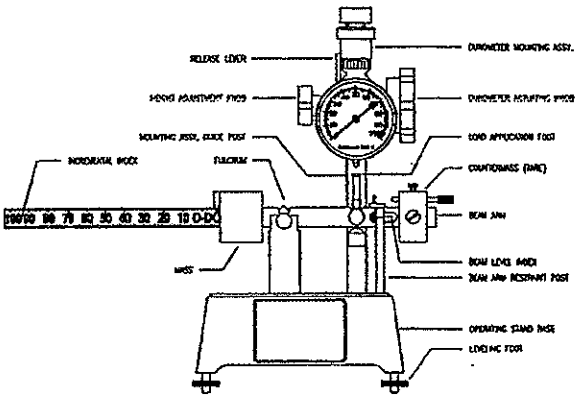


FIG. 5 Example of Durometer Calibration Apparatus

7.4 *Spring Calibration*—The durometer spring shall be calibrated at displayed readings of 10, 20, 30, 40, 50, 60, 70, 80, and 90. The measured force (9.8× mass in kilograms) shall be within the spring calibration tolerance specified in Table 1. Table 1 identifies the measured force applied to the indentor for the entire range of the instrument, although it is necessary only to verify the spring calibration at points listed herein.

7.5 *Spring Calibration Procedure*:

7.5.1 Ensure that the indentor extension has been adjusted in accordance with 7.1, and the linear relationship between indentor travel and display is as specified in 7.2.

7.5.2 Place the durometer in the calibration device as depicted in Fig. 5. Apply the forces indicated in Table 1 so that forces applied are aligned with the centerline of the indentor in a fashion that eliminates shock or vibration and adjust the durometer according to manufacturers' recommendations so that:

7.5.3 At the points enumerated in 7.4, the display shall indicate a value equal to 0.025 mm (0.001 in.) of indentor travel. For Type OOO-S durometers, the display shall indicate a value equal to 0.05 mm (0.002 in.) of indentor travel. For Type M durometers, the display shall indicate a value equal to 0.0125 mm (0.0005 in.) of indentor travel within the spring calibration tolerances specified in 7.6.

7.6 Spring calibration tolerances are ±1.0 durometer units for Types A, B, C, D, E, O, and DO, ±2.0 durometer units for Types OO, OOO, and OOO-S, and ±4.0 durometer units for Type M, while not indicating below 0 or above 100 at the time of calibration (see Table 1).

7.7 *Spring Force Combinations*:

7.7.1 For Type A, B, E, and O durometers:

Force, N = 0.55 + 0.075 HA

Where HA = hardness reading on Type A, B, E, and O durometers.

7.7.2 For Type C, D, and DO durometers:

Force, N = 0.4445 HD

Where HD = hardness reading on Type C, D, and DO durometers.

7.7.3 For Type M durometers:

Force, N = 0.324 + 0.0044 HM

Where HM = hardness reading on Type M durometers.

7.7.4 For Type OO and OOO durometers:

Force, N = 0.203 + 0.00908 HOO

Where HOO = hardness reading on Type OO durometers.

7.7.5 For Type OOO-S durometers:

Force, N = 0.167 + 0.01765 HOOO-S

Where HOOO-S = hardness reading on Type OOO-S durometers.

7.8 The rubber reference block(s) provided for verifying durometer operation and state of calibration are not to be relied upon as calibration standards. The calibration procedures outlined in Section 7 are the only valid calibration procedures.

7.8.1 The use of metal reference blocks is no longer recommended (see Note 2).

7.9 Verifying the state of durometer calibration, *during routine use*, may be accomplished by:

7.9.1 Verifying that the zero reading is no more than 1 indicated point above zero, and not below zero (on durometers so equipped), when the durometer is positioned so that no external force is placed upon the indentor.

7.9.2 Verifying that the 100 reading is no more than 100 and no less than 99 when the durometer is positioned on a flat surface of a non-metallic material so that the presser foot is in complete contact, causing the indentor to be fully retracted.

7.9.2.1 It is important that when performing the verification of 100, as described in 7.9.2, that extreme care be taken so as to not cause damage to the indentor. Verification of the 100 value is not recommended for durometers having a spring force greater than 10 N (Types C, D, and DO).

7.9.2.2 When performing the verification of 100, as described in 7.9.2, the non-metallic material shall be of a hardness value greater than 100 of the type (scale) of the durometer being employed. Tempered glass of a thickness greater than 6.35 mm (0.25 in.) has been found satisfactory for this application.

7.9.3 Verifying the displayed reading at any other point using commercially available rubber reference blocks which are certified to a stated value of the type (scale) of the durometer being employed. The displayed value of the durometer should be within ±2 durometer points of the reference block's stated value.

7.9.4 Verification of the zero and 100 readings of a durometer provide reasonable assurance that the linear relationship between the indicated display and the durometer mechanism remain valid.

7.9.5 Verification of points between zero and 100 provide reasonable assurance that the curvilinear relationship between the indicated display and the durometer mechanism remain valid.

7.9.6 *This is not a calibration procedure, it is a means by which a user may routinely verify that the durometer may be functioning correctly.* (See Note 2.)

8. Laboratory Atmosphere and Test Specimen Conditioning

8.1 Tests shall be conducted in the standard laboratory atmosphere, as defined in Practice D 618, Section 4.2.

8.2 The instrument shall be maintained in the standard laboratory atmosphere, as defined in Practice D 618, Section 4.1, for 12 h prior to performing a test.

8.3 The specimen shall be conditioned in accordance with condition 40/23 exclusive of humidity control, as described in Practice D 618, Section 8.1, Procedure A and tested under the same conditions, exclusive of humidity control.

8.4 These procedures may be modified if agreed upon between laboratories or between supplier and user and are in accordance with alternative procedures identified in Practice D 618.

8.5 No conclusive evaluation has been made on durometers at temperatures other than 23.0 ± 2.0°C (73.4 ± 3.6°F). Conditioning at temperatures other than the above may show changes in calibration. Durometer use at temperatures other than the above should be decided locally (see Practice D 1349).

9. Procedure

9.1 *Operating Stand Operation (Type 3 Operating Stand Required for Type M)*:

9.1.1 Care shall be exercised to minimize the exposure of the instrument to environmental conditions that are adverse to the performance of the instrument, or adversely affect test results.

9.1.2 Adjust the presser foot to support table parallelism as described in 5.1.2.1. It is necessary to make this adjustment each time the support table is moved to accommodate specimens of varying dimensions.

9.1.3 Prior to conducting a test, adjust the vertical distance from the presser foot to the contact surface of the test specimen to 25.4 ± 2.5 mm (1.00 ± 0.100 in.), unless it is known that identical results are obtained with presser foot at a greater or lesser vertical distance from the test specimen contact surface, or if otherwise stipulated by the manufacturer.

9.1.4 Place the specimen on the specimen support table, in a manner that the contact point of the indentor is in accordance with Section 6, unless it is known that identical results are obtained when measurements are made with the indentor at a lesser distance from the edge of the test specimen.

9.1.5 Actuate the release lever (Fig. 2) of the operating stand or activate the electromechanical device, allowing the durometer to descend at a controlled rate and apply the presser foot to the specimen in accordance with 5.1.2. In the case of "specimen to indentor" type operating stands, operate the lever or other mechanism to apply the specimen to the indentor in a manner that assures parallel contact of the specimen to the durometer presser foot without shock and with just sufficient force to overcome the calibrated spring force as shown in Table 1.

9.1.6 An operating stand that applies the mass at a controlled rate of descent, without shock is mandatory for Type M durometers. Hand-held application or the use of a Type 1 or Type 2 operating stand for the Type M durometer is not an acceptable practice, see 5.1.2.4.

9.1.7 For any material covered in 1.1, once the presser foot is in contact with the specimen, for example, when the initial indentor travel has ceased, the maximum indicated reading shall be recorded. The time interval of 1 s, between initial indentor travel cessation and the recording of the indicated reading, shall be considered standard. Other time intervals, when agreed upon among laboratories or between supplier and user, may be used and reported accordingly. The indicated hardness reading may change with time.

9.1.7.1 If the durometer is equipped with an electronic maximum indicator or timing device (refer to 5.1.1.9) the indicated reading shall be recorded within 1 ± 0.3 s of the cessation of indentor travel and reported (refer to 10.2.9 for reporting protocols), unless otherwise noted.

9.1.7.2 If the durometer is equipped with an analog type maximum indicator (refer to 5.1.1.10), the maximum indicated reading may be recorded and shall be reported (refer to 10.2.9), unless otherwise noted.

9.1.7.3 If the durometer is not equipped with the devices described in 5.1.1.9 or 5.1.1.10, the indicated reading shall be recorded within 1 s as is possible and reported (refer to 10.2.9), unless otherwise noted.

9.1.8 Make five determinations of hardness at different positions on the specimen at least 6.0 mm (0.24 in.) apart, 0.80 mm (0.030 in.) apart for Type M; and calculate the arithmetic mean, or alternatively calculate the median. The means of calculating the determinations shall be reported according to 10.2.8

9.2 *Manual (Hand Held) Operation of Durometer*:

9.2.1 Care shall be exercised to minimize the exposure of the instrument to environmental conditions that are adverse to the performance of the instrument, or adversely affect test results.

9.2.2 Place the specimen on a flat, hard, horizontal surface. Hold the durometer in a vertical position with the indentor tip at a distance from any edge of the specimen as described in Section 6, unless it is known that identical results are obtained when measurements are made with the indentor at a lesser distance.

9.2.3 Apply the presser foot to the specimen, maintaining it in a vertical position keeping the presser foot parallel to the specimen, with a firm smooth downward action that will avoid shock, rolling of the presser foot over the specimen, or the application of lateral force. Apply sufficient pressure to assure firm contact between the presser foot and the specimen.

9.2.4 For any material covered in 1.1, after the presser foot is in contact with the specimen, the indicated reading shall be recorded within 1 ± 0.1 s, or after any period of time agreed upon among laboratories or between supplier and user. If the durometer is equipped with a maximum indicator, the maximum indicated reading shall be recorded within 1 ± 0.1 s of the cessation of initial indentor travel. The indicated hardness reading may change with time.

9.2.5 Make five determinations of hardness at different positions on the specimen at least 6.0 mm (0.24 in.) apart and calculate the arithmetic mean, or alternatively calculate the median. The means of calculating the determinations shall be reported according to 10.2.8.

9.3 It is acknowledged that durometer readings below 20 or above 90 are not considered reliable. It is suggested that readings in these ranges not be recorded.

9.4 Manual operation (handheld) of a durometer will cause variations in the results attained. Improved repeatability may be obtained by using a mass, securely affixed to the durometer and centered on the axis of the indentor. Recommended masses

TABLE 2 Type 1 Precision—Type M Durometer Method

Material		Within Laboratories			Between Laboratories		
	MEAN	Sr^A	r^B	$(r)^C$	SR^D	R^E	$(R)^F$
1	31.8	1.26	3.58	11.24	3.76	10.63	33.41
2	40.8	1.14	3.23	7.90	2.47	7.00	17.13
3	54.0	0.975	2.76	5.11	2.38	6.73	12.46
4	62.8	0.782	2.21	3.52	2.24	6.34	10.10
5	70.9	0.709	2.01	2.83	0.974	2.76	3.89
6	80.6	1.686	4.77	5.92	1.61	4.56	5.65
7	87.7	1.15	3.25	3.71	2.63	7.45	8.50
8	32.4	0.947	2.68	8.26	3.64	10.29	31.73
9	41.8	0.797	2.26	5.40	2.23	6.31	15.11
10	53.3	0.669	1.89	3.55	2.29	6.49	12.17
11	63.2	0.485	1.37	2.17	2.19	6.20	9.80
12	69.6	0.737	2.09	3.00	0.99	2.80	4.02
13	78.3	0.784	2.22	2.84	1.04	2.94	3.75
14	87.6	1.121	3.17	3.62	2.65	7.49	8.55
15	34.1	0.85	2.40	7.05	1.84	5.20	15.25
16	42.3	0.635	1.80	4.25	1.20	3.39	8.01
17	54.6	0.56	1.59	2.90	2.15	6.09	11.15
18	62.9	1.12	3.17	5.04	1.47	4.16	6.61
19	70.3	0.689	1.95	2.77	0.944	2.67	3.80
20	81.7	0.483	1.37	1.67	1.10	3.10	3.80
21	87.9	0.879	2.49	2.83	2.07	5.86	6.67
AVERAGE	61.4						
POOLED VALUES		0.924	2.62	4.26	2.146	6.07	9.89

^{A} Sr = repeatability standard deviation, measurement units.
^{B} r = repeatability = 2.83 × Sr, measurement units.
^{C} (r) = repeatability, relative, (that is, in percent).
^{D} SR = reproducibility standard deviation, measurement units.
^{E} R = reproducibility = 2.83 × SR, measurement units.
^{F} (R) = reproducibility, relative, (that is, in percent).

are 1 kg for Type A, B, E, and O durometers, 5 kg for Type C, D, and DO durometers, and 400 g for Type OO, OOO, and OOO-S durometers. The introduction of an additional mass on Type M durometers is not permitted. Further improvement may be achieved by the use of a durometer operating stand that controls the rate of descent of the durometer presser foot to the test specimen and incorporates the masses described above.

10. Report

10.1 *Instrument Calibration Report (Durometer or Operating Stand)*:

10.1.1 Date of calibration.

10.1.2 Date of last calibration.

10.1.3 Calibration due date (see Note 2).

10.1.4 Manufacturer, type, model, and serial number of the instrument, and a notation when a maximum indicator or timing device is present.

10.1.5 Values obtained (pre- and post-calibration results), including a notation of the effect of a maximum indicator, if present. The method of reporting the calibrated value shall be by attaining the arithmetic mean of the determinations.

10.1.6 Ambient temperature.

10.1.7 Relative humidity.

10.1.8 Technician identification.

10.1.9 Applicable standards to which the instrument is calibrated.

10.1.10 Calibrating instrument information to include type, serial number, manufacturer, date of last calibration, calibration due date (see Note 2), and a statement of traceability of standards used to NIST or other acceptable organization. See 1.4.

TABLE 3 Type 1 Precision—Type A Durometer Method

Material	Average Level	Within Laboratories			Between Laboratories		
		Sr^A	r^B	$(r)^C$	SR^D	R^E	$(R)^F$
1	51.4	0.646	1.83	3.56	1.56	4.41	8.59
2	65.3	0.878	2.48	3.81	2.21	6.06	9.27
3	68.0	0.433	1.23	1.80	2.28	6.45	9.49
Pooled	61.6	0.677	1.92	3.11	2.018	5.72	9.28

^{A} Sr = repeatability standard deviation, measurement units.
^{B} r = repeatability = 2.83 × Sr, measurement units.
^{C} (r) = repeatability, relative, (that is, in percent).
^{D} SR = reproducibility standard deviation, measurement units.
^{E} R = reproducibility = 2.83 × SR, measurement units.
^{F} (R) = reproducibility, relative, (that is, in percent).

TABLE 4 Type 1 Precision—Type D Durometer Method

Material	Average Level	Within Laboratories			Between Laboratories		
		Sr^A	r^B	$(r)^C$	SR^D	R^E	$(R)^F$
1	42.6	0.316	0.894	2.10	2.82	7.98	18.7
2	54.5	0.791	2.24	4.11	3.54	10.0	18.4
3	82.3	1.01	2.86	3.47	3.54	10.0	12.2
Pooled	59.8	0.762	2.16	3.61	3.32	9.40	15.7

^{A} Sr = repeatability standard deviation, measurement units.
^{B} r = repeatability = 2.83 × Sr, measurement units.
^{C} (r) = repeatability, relative, (that is, in percent).
^{D} SR = reproducibility standard deviation, measurement units.
^{E} R = reproducibility = 2.83 × SR, measurement units.
^{F} (R) = reproducibility, relative, (that is, in percent).

10.2 *Hardness Measurement Report:*

10.2.1 Date of test.

10.2.2 Relative humidity.

10.2.3 Ambient temperature.

10.2.4 Manufacturer, type, and serial number of the durometer or operating stand, or both, including a notation when a maximum indicator or timing device is present, date of last calibration, and calibration due date (see Note 2).

NOTE 2—The calibration interval (calibration due date) for a durometer is to be determined by the user, based upon frequency of use, severity of conditions, environmental factors, and other variables.

Periodic checking of the operation and state of durometer calibration using commercially available rubber test blocks (refer to 7.8), specifically designed for this purpose, is recommended.

An instrument that has been exposed to severe shock, is visibly damaged, produces test determinations more than 2 points different from calibrated rubber test blocks or other reference standard, or is otherwise suspected of unreliability, should be removed from service and returned to a qualified calibration facility.

A calibration interval of one year is recommended for durometer test blocks and durometer instruments that are infrequently used, more often for others.

The calibration interval for instruments and peripheral devices employed in the calibration of durometers is to be determined by the calibration service provider. It is recommended that the protocols outlined in ISO/IEC 17025, as required by the manufacturer, and those to which the service is provided, be followed.

10.2.5 Means of testing, whether manual (hand held), Type 1 operating stand (specimen to indentor), Type 2 operating stand (indentor to specimen type), or Type 3 operating stand (electromechanical or hydraulically dampened).

10.2.6 Description of test specimen, including thickness, number of pieces plied if less than the thickness indicated in Section 6, including the vulcanization date.

10.2.7 Complete identification of material tested.

10.2.8 Hardness value obtained and method of calculation, either arithmetic mean or alternatively, the median.

10.2.9 Indentation hardness time interval at which determination was made. Readings may be reported in the form: M/60/1 where M is the type of durometer, 60 the reading, and 1 the time in seconds that the presser foot is in contact with the specimen or from an electronic timing device.

11. Precision and Bias

11.1 These precision and bias statements have been prepared in accordance with Practice D 4483. Refer to this Practice for terminology and other testing and statistical concepts.

11.2 The Type 1 precision for the Type M method was determined from an interlaboratory program with 21 materials of varying hardness, with six participating laboratories. Tests were conducted on two separate days in each laboratory for the Type M testing program. All materials were supplied from a single source, being those commonly supplied as reference materials with the instruments from the manufacturer.

11.3 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers) used in the particular interlaboratory program as described above. The precision parameters should not be used for acceptance or rejection testing, or both, of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

11.4 The Type 1 precision for both Type A and D methods was determined from an interlaboratory program with 3 materials of varying hardness, with six participating laboratories. Tests were conducted on two separate days in each laboratory for both A and D testing programs. All materials were supplied from a single source.

11.5 A test result for hardness, for Types A, D, and M, was the median of five individual hardness readings on each day in each laboratory.

11.6 Table 2 shows the precision results for Type M method,[4] Table 3 shows the precision results for Type A method,[5] and Table 4 gives the precision results for Type D method.[5]

11.7 *Precision*—The precision of this test method may be expressed in the format of the following statements which use as appropriate value r, R, (r), or (R), that is, that value to be used in decisions about test results (obtained with the test method). The appropriate value is that value of r or R associated with a mean level in Table 1 closest to the mean level under consideration (at any given time, for any given material) in routine testing operations.

NOTE 3—A Type 1 precision statement for Types E, OOO, OOO-S, and R have not yet been made available.

11.7.1 *Repeatability*—The repeatability, r, of these test methods has been established as the appropriate value tabulated in Tables 2-4. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated r (for any given level) must be considered as derived from different or non-identical sample populations.

11.7.2 *Reproducibility*—The reproducibility, R, of these test methods has been established as the appropriate value tabulated in Tables 2-4. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated R (for any given level) must be considered to have come from different or non-identical sample populations.

11.7.3 Repeatability and reproducibility are expressed as a percentage of the mean level, (r) and (R), and have equivalent application statements as above for r and R. For the (r) and (R) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

11.8 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by this test method. Bias, therefore cannot be determined.

12. Keywords

12.1 durometer; durometer hardness; hardness; indentation hardness; micro durometer hardness

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D11-1091.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D11-1029.

APPENDIXES

(Nonmandatory Information)

X1. DUROMETER SELECTION GUIDE

X1.1 The durometer selection guide is designed to assist in the selection of the proper durometer type for various applications.

X1.2 It is generally recognized that durometer hardness determination below 20 and above 90 are unreliable. It is recommended that the next lower or higher type (scale) be used in these situations.

X1.3 It is also recommended that, whenever possible, an operating stand be employed in performing durometer hardness tests.

TABLE X1.1 Durometer Selection: Typical Uses

Type (Scale)	Typical Examples of Materials Tested	Durometer Hardness (Typical Uses)
A	Soft vulcanized rubber, natural rubber, nitriles, thermoplastic elastomers, flexible polyacrylics and thermosets, wax, felt, and leathers	20–90 A
B	Moderately hard rubber, thermoplastic elastomers, paper products, and fibrous materials	Above 90 A Below 20 D
C	Medium-hard rubber, thermoplastic elastomers, medium-hard plastics, and thermoplastics	Above 90 B Below 20 D
D	Hard rubber, thermoplastic elastomers, harder plastics, and rigid thermoplastics	Above 90 A
DO	Moderately hard rubber, thermoplastic elastomers, and very dense textile windings	Above 90 C Below 20 D
M	Thin, irregularly shaped rubber, thermoplastic elastomer, and plastic specimens	20–85 A
O	Soft rubber, thermoplastic elastomers, very soft plastics and thermoplastics, medium-density textile windings	Below 20 DO
OO	Extremely soft rubber, thermoplastic elastomers, sponge, extremely soft plastics and thermoplastics, foams, low-density textile windings, human and animal tissue	Below 20 O
CF	Composite foam materials, such as amusement ride safety cushions, vehicle seats, dashboards, headrests, armrests, and door panels	See Test Method F 1957

X2. RELATED TEST METHODS[2]

C 367 Test Methods for Strength Properties of Prefabricated Architectural Acoustical Tile or Lay-In Ceiling Panels

C 473 Test Methods for Physical Testing of Gypsum Panel Products

C 581 Practice for Determining Chemical Resistance of Thermosetting Resins Used in Glass-Fiber-Reinforced Structures Intended for Liquid Service

C 661 Test Method for Indentation Hardness of Elastomeric-Type Sealants by Means of a Durometer

C 836 Specification for High Solids Content, Cold Liquid-Applied Elastomeric Waterproofing Membrane for Use with Separate Wearing Course

D 461 Test Methods for Felt

D 531 Test Method for Rubber Property—Pusey and Jones Indentation

D 619 Test Methods for Vulcanized Fibre Used for Electrical Insulation

D 1037 Test Methods for Evaluating Properties of Wood-Base Fiber and Particle Panel Materials

D 1054 Test Method for Rubber Property—Resilience Using a Goodyear-Healey Rebound Pendulum

D 1414 Test Methods for Rubber O-Rings

D 1474 Test Methods for Indentation Hardness of Organic Coatings

D 2134 Test Method for Determining the Hardness of Organic Coatings with a Sward-Type Hardness Rocker

D 2287 Specification for Nonrigid Vinyl Chloride Polymer and Copolymer Molding and Extrusion Compounds

D 2583 Test Method for Indentation Hardness of Rigid Plastics by Means of a Barcol Impressor

D 2632 Test Method for Rubber Property—Resilience by Vertical Rebound

D 4289 Test Method for Elastomer Compatibility of Lubricating Greases and Fluids

D 5672 Test Method for Flexible Cellular Materials Measurement of Indentation Force Deflection Using a 25-mm (1-in.) Deflection Technique

D 6546 Test Methods for and Suggested Limits for Determining Compatibility of Elastomer Seals for Industrial Hydraulic Fluid Applications

F 1151 Test Method for Determining Variations in Hardness of Film Ribbon Pancakes

NOTE X2.1—The hardness testing of other nonmetallic materials may be under the jurisdiction of one or more ASTM committees; the respective committee should be contacted for specific information.

EXHIBIT 4

REDACTED

EXHIBIT 5

REDACTED

EXHIBIT 6

REDACTED

EXHIBIT 7

REDACTED

EXHIBIT 8

REDACTED

EXHIBIT 9

REDACTED

EXHIBIT 10

REDACTED

EXHIBIT 11

REDACTED

EXHIBIT 12

REDACTED

EXHIBIT 13

REDACTED

EXHIBIT 14

REDACTED